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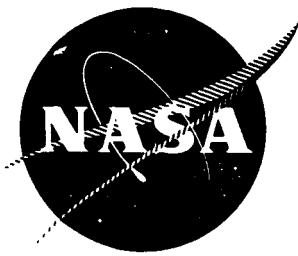
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**EVALUATION OF LOW COST/HIGH TEMPERATURE INSULATION**

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**MARTIN MARIETTA AEROSPACE  
DENVER, COLORADO**

**NOVEMBER 1975**



## EVALUATION OF LOW COST/HIGH TEMPERATURE INSULATION

by E. L. Strauss

**MARTIN MARIETTA AEROSPACE**  
Denver, Colorado

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16. Abstract  Six fiber products and six insulation blankets comprising silica, alumina, zirconia, mullite, and mixed ceramic systems were subjected to furnace exposures up to 500 hours at temperatures of 1000° to 1600°C and evaluated for chemical and dimensional stability and for changes in thermal conductivity. Alumina, zirconia, and mullite fibers were fabricated into Reusable Surface Insulation (RSI) tile by water-felting and reimpregnation with ethyl silicate. Specimens were exposed to 25 thermal cycles at 1200°C and 1400°C and a pressure of 10 and 32 torr, respectively. Production costs for 930 m <sup>2</sup> (10,000 ft <sup>2</sup> ) of blanket insulation and of alumina RSI tile were developed.		
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## **FOREWORD**

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This report was prepared by the Denver Division, Martin Marietta Aerospace under Contract NAS3-18900 for the Lewis Research Center, National Aeronautics and Space Administration. The work was administered under the direction of the Lewis Research Center's Materials and Structures Division with Mr. John P. Merutka acting as Project Manager.

The program was conducted by the Structures and Materials Department of the Engineering, Research and Technology organization during the period from 25 June 1974 to 30 June 1975. Mr. Eric L. Strauss of Martin Marietta Aerospace was the Program Manager and was also responsible for its technical direction.

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## SUMMARY

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Twelve oxide fiber products comprising bulk fibers and blanket insulations and including silica, alumina, stabilized zirconia, mullite, silica-chromia, alumina-silica, and alumina-silica-chromia systems were evaluated for chemical and dimensional stability and for changes in thermal conductivity when exposed up to 500 hours to temperatures of 1000, 1200, 1400, and 1600°C in gas-fired and/or electric kilns. Fibers were tested in the form of water-felted fiber cakes while blanket insulations were cut into specimens of the appropriate size. Chemical stability was determined by periodic visual examination and by scanning electron microscopy and X-Ray diffraction before and after exposure. Thermal conductivity of blanket insulations was measured at room temperature by the line source technique. Dimensional stability was determined by physical weighing and measuring before exposure and after 25, 100 and 500 cumulative hours at temperature.

The time-temperature limits for satisfactory performance of fiber cakes and blankets (based on changes in density and linear dimensions) were established and are tabulated. In this study, a density increase greater than 30% or a linear shrinkage greater than 15% denotes unsatisfactory performance. All felted fiber cakes and three out of six blankets are rated satisfactory for 500 hours at 1000°C. None of the materials had satisfactory performance after 25 hours at 1600°C.

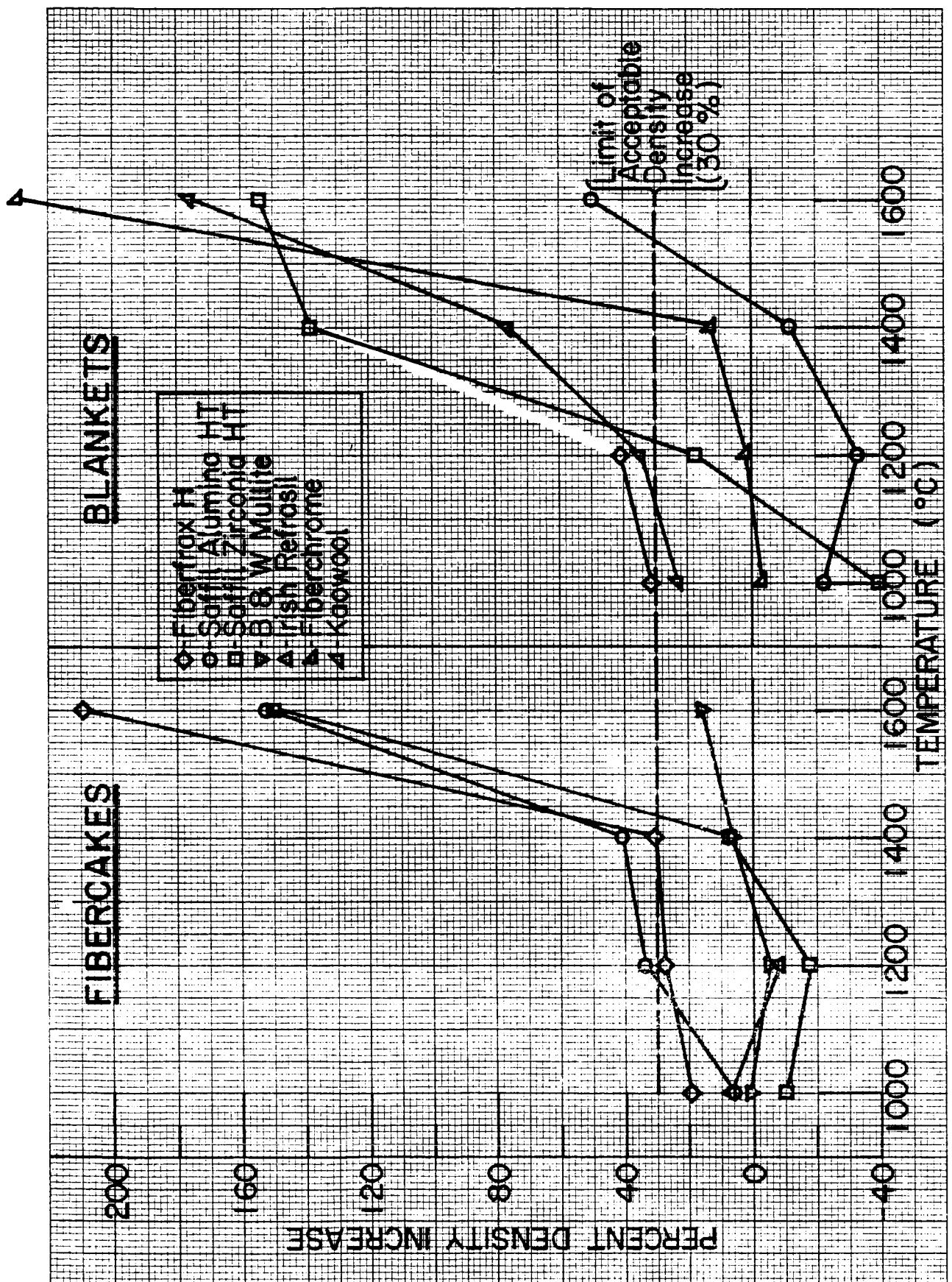
Density of most blankets and fiber cakes increased sharply between 1400 and 1600°C as illustrated by the plot of 100-hour exposure data.

Saffil Alumina HT fiber cakes exhibited unacceptable shrinkage above 1000°C. This was attributed to an improperly processed fiber batch and a replacement batch exhibited good dimensional stability at 1400°C. Saffil Zirconia HT fiber cakes puffed up at 1000 and 1200°C but became rigid at 1400°C. The B&W Mullite cakes had significant weight loss at 1400°C yet exhibited good dimensional stability. The 3 Micron Mullite fiber cakes were fragile and tended to break when handled. Fiberfrax H fiber cakes had high densities due to shrinkage during drying and thickness decreased further during high temperature exposure. Irish Refrasil cakes remained noncrystalline after 1000°C exposure but exhibited strong crystobalite peaks after 100 hours at 1200°C.

*Time-Temperature Limits for Satisfactory Performance of Fiber Cakes and Blanket Insulations*

Material	1000°C			1200°C			1400°C		
	Density Change	Dimensional Stability	Dimensional Stability						
<u>Fibercakes</u>									
Irish Refrasil	500 hr	500 hr	500 hr	500 hr	500 hr	500 hr	No test	No test	No test
Saffil Alumina HT*	500 hr	500 hr	< 25 hr	500 hr	< 25 hr	500 hr	< 25 hr	500 hr	500 hr
Saffil Zirconia HT	500 hr	500 hr	500 hr	500 hr	500 hr	500 hr	100 hr	100 hr	100 hr
B&W Mullite	500 hr	500 hr	500 hr						
3 Micron Mullite	500 hr	500 hr	500 hr	500 hr	500 hr	500 hr	25 hr	25 hr	25 hr
Fiberfrax H	500 hr	500 hr	500 hr	500 hr	25 hr	25 hr	< 25 hr	< 25 hr	< 25 hr
<u>Blankets</u>									
Fiberfrax H	< 25 hr	< 25 hr	25 hr	25 hr	25 hr	25 hr	No test	No test	No test
Kaowool 1400	500 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr
Fiberchrome	500 hr	500 hr	500 hr	500 hr	500 hr	500 hr	100 hr	100 hr	100 hr
Saffil Alumina HT	500 hr	500 hr	500 hr						
Saffil Zirconia HT	500 hr	500 hr	100 hr	25 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr
Microquartz	< 25 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr	< 25 hr	No test	No test	No test

\*Data is for old fiber lot. Performance of new fiber lot can be extrapolated to be satisfactory for 500 hours at 1400°C.



Density Changes in Fiber Cakes and Blankets After 100-hour Exposure to Various Temperatures

Saffil Alumina HT blanket contained an organic binder that resulted in an initial weight loss of 10%. Although the samples puffed up in thickness, the length and width were stable to 1400°C. Saffil Zirconia HT blanket also contained an organic binder whose decomposition resulted in a 7% weight loss. Samples puffed up at 1000°C but became semirigid at 1200°C. Kaowool 1400 blanket shrank in thickness at all temperatures but length, width and weight were stable to 1200°C. Fiberchrome blanket had good dimensional stability to 1400°C despite some loss of chromia. Microquartz and Fiberfrax H blankets tended to harden, twist and deform at 1000 and 1200°C. A high temperature heat treatment would stabilize these fibers and prevent deleterious shrinkage in service.

Crystallographic examination showed no changes as a result of 1000°C exposures, but all materials exhibited grain growth after 500 hours at 1400°C. Crystallinity of all fibers increased with exposure temperature.

Saffil Alumina HT and Fiberchrome blankets had satisfactory performance at 1400°C for 500 hours and 100 hours, respectively. These blankets plus Kaowool 1400 (whose unsatisfactory dimensional stability was due to shrinkage in thickness) were tested to determine the temperatures (500 hour exposure) at which increases in thermal conductivity become unacceptable. In this study, a conductivity increase greater than 10% denotes unsatisfactory performance. All blankets had sharply increased conductivities after the 1400°C exposure. The conductivity increase of Kaowool 1400 exposed at 1000 and 1200°C can be correlated with its increased density.

*Temperature Limit for Satisfactory Thermal Conductivity of Oxide Fiber Blankets*

Material	Temperature Limit, 500 hour exposure
Saffil Alumina HT	1200°C
Kaowool 1400	Less than 1000°C
Fiberchrome	1200°C

Saffil Alumina HT, Saffil Zirconia HT, and B&W Mullite were chosen as the fiber systems for 0.15 g/cc (9 lb/ft<sup>3</sup>) RSI (Reusable Surface Insulation) development because they exhibited good thermal stability during furnace testing and felted fiber cakes of the proper density could be readily produced. Ethyl silicate (Stauffer Silbond H-4) was selected as the binder. It was incorporated into the fiber cakes by secondary impregnation of an isopropyl alcohol solution and gelled *in situ* by adding NH<sub>4</sub>OH to adjust its pH. RSI tiles were fired for 30 minutes at 1371°C (2500°F) and exhibited a linear firing shrinkage of less than 2-1/2%.

RSI specimens were thermally cycled 25 times at surface temperatures of 1200 and 1400°C (2192 and 2552°F) and pressures of 10 and 32 torr respectively. Flexural properties, room temperature thermal conductivity, chemical stability (scanning electron microscopy and X-Ray diffraction), dimensional stability and weight loss were determined for exposed specimens and unexposed controls. Weight and dimensions did not change significantly as a result of thermal/vacuum exposure and crystallinity remained unchanged. Room temperature flexural properties and thermal conductivity were not degraded.

Material and fabrication costs were determined for 930 m<sup>2</sup> (10,000 ft<sup>2</sup>) of blanket insulation and a like amount of 2.5 cm (1 in.) thick alumina RSI tile. Blanket material costs were about \$12,700 for Kaowool 1400 and Fiberchrome and \$38,400 for Saffil Alumina HT. Material and fabrication costs were \$513,130 for Alumina RSI tile or \$21.38 per individual tile 20.3x20.3x2.5 cm (8x8x1 in.) in size. Raw material costs for these fibers and blankets are less than \$30 per kg. This price is considered "low cost" for aerospace use.

To select the optimum fiber, blanket, or RSI material the requirements of the intended application must be considered. The relative importance of such factors as cost, density, temperature resistance, thermal stability, strength and elongation will determine which material system can provide the best overall performance.

## I. INTRODUCTION

---

Industry-wide development of high-temperature oxide fibers and their processing and evaluation as reusable surface insulation (RSI) tiles has all but ceased since the Lockheed silica RSI material (based on Johns-Manville's Micro-Quartz fibers) was selected for Shuttle orbiter external thermal protection. While the silica material offers the advantages of low density, low thermal conductivity, and low thermal expansion, its temperature capability is limited by the conversion of the silica fiber and binder from the vitreous form to crystobalite, which is accompanied by significant shrinkage. Other fiber types including silica-chromia, alumino silicate, mullite, alumina, zirconia, and zirconia silicate, are available either as commercial products or as specialty laboratory items, but their overall potential has never been evaluated in a systematic manner. In some instances (e.g., mullite fibers) fiber production has been discontinued, but a quantity of fibers is still available from earlier efforts. A program to establish the aerospace potential of these fiber systems, while limited supplies are still available and the technical know-how is still current, is therefore very necessary and timely.

The objective of this program was to establish the time-temperature limits below which 0.15g/cc (9 lb/ft<sup>3</sup>) reusable surface insulation tile and 0.13 g/cc (8 lb/ft<sup>3</sup>) flexible blanket insulation can withstand repeated thermal cycles simulating reentry without experiencing physical deterioration, linear shrinkage greater than 15%, density increases greater than 30%, or thermal conductivity increases of more than 10%. The temperature range of interest was 1000 to 1600°C (1832 to 2912°F). The technical approach was designed to yield comparative data on fiber crystallography, and material density, strength, and thermal conductivity of unexposed controls and thermally exposed specimens.

The technical effort was divided into two sequential tasks:

Task I - Thermal Exposure and Evaluation of Water-Felted Fiber Cakes and Blanket Insulations;

Task II - RSI Tile Manufacture, Thermal Exposure, and Evaluation.

## II.

FIBER AND BLANKET MATERIALS

---

Six oxide fiber products and six blanket insulation systems were procured for this program. Table 1 describes the 12 materials and their suppliers and unit costs. Additional data on the materials systems are contained in vendor data (refs. 1 through 6). The materials range from fully commercial systems (Fiberfrax H, Microquartz, Fiberchrome, Kaowool) to new commercial fibers (Saffil Alumina and Zirconia HT), to experimental fibers (Mullite, Irish Refrasil type SS-19-A5).

Fiber diameter and shot content of the six fiber products were determined by scanning electron microscopy at magnification of approximately 330X and 1000X, using a Cambridge high-resolution SEM unit. The fiber samples were gold-plated to accentuate the individual fibers and shot particles. Representative SEM photographs are shown in Figures 1 through 6. The photographs show an uniform fiber diameter for Irish Refrasil, Saffil Alumina HT, and Saffil Zirconia HT. Fiberfrax H and the Mullite samples show a wide dispersion of fiber diameters. No shot particles were observed in the Irish Refrasil and Saffil Alumina HT samples and only a single isolated shot particle was noted in Saffil Zirconia HT. Fiberfrax H and the two Mullite materials contained significant shot concentrations. A summary of fiber diameter and shot content data is presented in Table 2.

To facilitate furnace testing of the fiber products, water-felted fiber cakes were produced. Thermal testing of the fiber cakes would indicate the suitability of the fibers for use in reusable surface insulation systems. The target density for the fiber cakes was 0.12 g/cc (7.5 lb/ft<sup>3</sup>), which would produce a 0.15 g/cc (9 lb/ft<sup>3</sup>) RSI tile after addition of a binder. Before felting, fiber bundles were dispersed and cleaned by using the technique described in NASA Tech Brief B73-10438 (ref. 7). Fiber clumps that were still held together by mechanical interlocking or by electrical charges were further dispersed by blending with distilled water in a Hobart mixer for 30 minutes. The amount of water used was 1000 cc per 25 g of fibers. Separan AP 30 deflocculating agent (ref. 8) was added to the water in a concentration of 0.05%. To dissolve Separan AP 30, the water was heated to near its boiling point and Separan AP 30 was added slowly, while the water was stirred vigorously. To felt 3 Micron Mullite fiber cakes, the alkalinity of the Separan AP 30 water solution was raised to pH-12 by addition of NH<sub>4</sub>OH.

Fiber cakes were felted to a thickness of approximately 2.54 cm (1 in.) in a rectangular 15.2x6.4 cm (6x2½ in.) felting rig equipped with a close fitting plunger. Excess water was drained from the felts by vacuum aspiration. After removal from the felting rig, the fiber cakes were dried at 65°C (150°F) in a circulating air oven.

Densities of the Irish Refrasil, Saffil Alumina HT, Saffil Zirconia HT, and B&W Mullite fiber cakes could be controlled close to the desired density of 0.12 g/cc. The Fiberfrax H and 3 Micron Mullite fiber cakes shrank during oven drying, resulting in a fiber cake density of approximately 0.18 g/cc (11.2 lb/ft<sup>3</sup>). The "as felted" 15.2x6.4 cm (6x2½ in.) fiber cakes were used as density/shrinkage specimens for furnace exposure testing. The 6.4x3.2 cm (2½x1¼ in.) specimens for scanning electron microscopy and X-Ray diffraction were cut from the larger cakes. Blanket specimens were cut from the as-received insulation blankets.

### III. EXPERIMENTAL PROCEDURES—FIBER CAKES AND BLANKETS

---

#### A. FURNACE EXPOSURE

Fiber cake and blanket specimens were heat soaked in a series of furnace runs conducted in the following sequence:

<u>Exposure Temperature</u>	<u>Exposure Time</u>	<u>Furnace</u>
1) 1000°C (1832°F)	500 hr	Gas-fired kiln
2) 1200°C (2192°F)	500 hr	Gas-fired kiln
3) 1400°C (2552°F)	500 hr	Gas-fired kiln
4) 1600°C (2912°F)	200 hr	Gas-fired kiln
5) 1000°C (1832°F)	100 hr	Electric kiln
6) 1200°C (2192°F)	100 hr	Electric kiln
7) 1400°C (2552°F)	25 hr	Electric kiln

Two gas-fired kilns were used in these tests: (1) a Lindberg propane-fired kiln of 1760°C (3200°F) peak temperature and 76x102 cm (30x40 in.) interior floor size; and (2) a natural gas-fired kiln of 1538°C (2800°F) peak temperature and a 94x99 cm (37x39 in.) interior floor size. The electric kiln was an ECS furnace of 1510°C (2750°F) peak temperature and a 30x46 cm (12x18 in.) interior floor size.

Individual fiber cakes and blankets were exposed for time periods listed in Table 3. The 3 Micron Mullite fibers, Saffil Alumina HT blanket, and Saffil Zirconia HT blanket were received after the start of furnace testing. The 3 Micron Mullite fiber cakes were introduced into the test sequence at 25 hours into the 1400°C gas-fired furnace run. Exposure data of 1000 and 1200°C for this material were obtained in the 100-hour electric kiln runs. The Saffil Alumina HT blanket was introduced into the test sequence at the beginning of the 1200°C gas-fired furnace run. The 1000°C exposure data were obtained in the 100-hour electric kiln run. The Saffil Zirconia HT blanket was introduced into the test sequence at 75 hours into the 1200°C gas-fired furnace run and measured after exposures of 25 and 425 hours. The 100-hour exposure data at 1000°C and 1200°C were obtained in electric kiln runs. The Saffil Alumina and Zirconia HT blankets were laboratory-produced samples supplied by ICI United States, Inc. After

completion of the test sequence, 10 lb each of a production lot of alumina and zirconia blanket were delivered. These materials were evaluated and compared with the earlier blanket samples by testing for 25 hours at 1400°C in the electric kiln. The two material lots were found to be equivalent.

Irish Refrasil fiber cake, Fiberfrax H blanket and Microquartz blanket were not tested beyond 100 hours at 1200°C since visual examination indicated that these materials would be unsuitable for more severe exposures. Irish Refrasil suffered extensive loss of chromia while Fiberfrax H and Microquartz blankets became rigid and deformed.

#### B. SPECIMEN CONFIGURATIONS

Three types of specimens were tested. They were 15.2x6.4x2.5 cm (6x2<sup>1</sup><sub>2</sub>x1 in.) density/shrinkage samples, 6.4x3.2x2.5 cm (2<sup>1</sup><sub>2</sub>x1<sup>1</sup><sub>4</sub>x1 in.) SEM/XRD samples, and 10.2x5.1x2.5 cm (4x2x1 in.) thermal conductivity samples (blanket materials only). Duplicate density/shrinkage and SEM/XRD specimens and four thermal conductivity samples were tested in each furnace run.

In the gas-fired furnace runs, the tests were interrupted and specimens inspected visually after 5, 15, 25, 50, 75, 100, 200, 350 and 500 hours of exposure. Density/shrinkage samples were weighted and measured before thermal testing and after 25, 100 and 500 hours of exposure. Materials exhibiting significant degradation were removed from the furnace. In the electric kiln runs, samples were visually inspected, weighed and measured after 25 and 100 hours of exposure.

#### C. VISUAL OBSERVATIONS

Visual observations of the fiber cake and blanket samples at periodic intervals during the test runs are summarized in Tables 4 through 8. Photographs of the density/shrinkage samples after exposure to the four test temperatures are shown in Figures 7 through 18.

#### IV.

#### SPECIMEN MEASUREMENTS AND MATERIAL CHARACTERIZATION

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##### A.

##### WEIGHT, DIMENSIONAL AND DENSITY CHANGES

Percent changes in size, weight, and density of samples after furnace exposure are listed in Tables 9 through 22. Plots of dimensional and weight change of fiber cakes and blankets as a function of exposure time at various temperatures are presented in Figures 19 through 34. Similar plots of 100-hour weight and dimensional change data as a function of exposure temperature are presented in Figures 35 through 46.

The Saffil Alumina HT fiber cake exhibited significant shrinkage after 25 hours at 1200°C and large differences were evident between the two duplicate samples (see Table 13). A third fiber cake sample was therefore introduced into the 1200°C furnace run after 50 hours. Its shrinkage was midway between the two original samples, confirming that large variations in shrinkage could be obtained with this batch of material. A sample of the alumina fiber was returned to ICI for their examination. They concluded that we had received an improperly processed batch and replaced it with a different lot of fiber. Fiber cakes felted from the new fiber were compared with the old fiber lot by testing for 25 hours at 1400°C in the electric kiln. The new fiber exhibited substantially less weight loss and shrinkage than the old fiber lot (see Table 20).

Fiberfrax H and Microquartz blankets became rigid and/or deformed during exposure in the gas-fired kiln at 1000 and 1200°C. Twisted specimens are difficult to measure and the tabulated dimensional change data are therefore not precise. Since the deformation in these materials can be affected by furnace gradients and heating rates, the blanket materials were retested in the electric kiln. Fiberchrome blanket was also retested at 1000°C and Kaowool 1400 was retested at 1000 and 1200°C in the electric kiln to determine the reproducibility of dimensional and weight change data for materials that remain flexible, flat, and square. Dimensional and weight change data of samples heated in the gas-fired and the electric kilns are compared in Table 23. Dimensional changes as determined in the two furnaces differed significantly for Fiberfrax H and Microquartz (materials which hardened and deformed), but differed by lesser amounts for the Kaowool 1400 and Fiberchrome samples that remained flat and square.

Tables 24 through 27 categorize the temperature-exposed fiber cakes and blanket materials by percent weight loss and dimensional change into three categories:

- 1) Increase or 0 to 2% decrease;
- 2) 2 to 6% decrease;
- 3) Greater than 6% decrease.

These summary charts are for 500 hours of exposure. Where specimens had been exposed for only 100 hours, the classification is based on an extrapolation to 500 hours. Note, however, that most materials had undergone a major portion of the measured change after 25 hours of exposure as illustrated by the plot of length change at 1200°C (Fig. 23).

#### B. SCANNING ELECTRON MICROSCOPY

Scanning electron micrographs at 1000X magnification were taken of controls and thermally exposed fiber cakes and blanket materials. SEM observations are summarized in Table 28. Photomicrographs are shown in Figure 47 through 56. In summary, no changes were observed in fiber morphology after exposure to 100 or 500 hours at 1000°C. The 1200°C exposure produced grain growth and fiber sintering in Saffil Alumina HT fiber cake (old fiber material) and some slight evidence of grain growth in Saffil Alumina HT blanket. Grain growth occurred in all fiber materials at 1400°C, ranging from moderate (3 Micron Mullite, Fiberfrax fiber-cake, Kaowool, and Saffil Alumina HT Blanket), to severe with fiber sintering or embrittlement (Saffil Alumina HT fiber cake, Saffil Zirconia HT fiber cake. Fiberchrome blanket, and Saffil Zirconia HT blanket). Severe grain growth with fiber fusion, sintering or embrittlement was observed in all materials after exposure at 1600°C.

#### C. X-RAY DIFFRACTION

X-Ray diffraction of controls and thermally exposed fiber cakes and blanket materials were obtained with a Norelco Model 12045 unit. Results are summarized in Table 29. Control samples were generally noncrystalline or exhibited weak crystalline peaks (Saffil Alumina HT; B&W and 3 Micron Mullite). Only Saffil Zirconia HT was strongly crystalline. After exposure at 1000°C, all materials except Irish Refrasil exhibited some crystallinity.

The Fiberfrax H blanket had moderately strong mullite peaks while the Saffil Zirconia HT fiber cake and blanket remained strongly crystalline. Crystallinity generally increased with increasing exposure temperature above 1000°C. Silica fibers (Irish Refrasil) contained crystobalite while silica-alumina fibers (Fiberfrax, Fiberchrome, Kaowool) exhibited mullite peaks and, in some instances, crystobalite peaks. The Mullite fiber cakes and Saffil Alumina HT fiber cakes and blankets exhibited mullite and alpha alumina peaks.

#### D. ROOM TEMPERATURE THERMAL CONDUCTIVITY

Room temperature thermal conductivity was measured by the line source technique (ref. 9) on three blanket materials, Saffil Alumina HT, Kaowool, and Fiberchrome. Conductivity was measured on unexposed controls and on samples exposed to 1000, 1200, and 1400°C. In the line source conductivity method, a heater wire and a thermocouple were placed inside a 10.2x5.1x5.1 cm (2x2x2 in.) sample. The samples were assembled from two halves, each 10.2x5.1x2.5 cm (4x2x1 in.) in size. A known amount of heat is put into the sample by passing a current through the heater wire, and the variation of temperature with time is measured with the thermocouple. Using the temperature-time history, the thermal conductivity is calculated by means of the equation:

$$k = \frac{q}{4\pi(\theta_2 - \theta_1)} \ln \frac{t_2}{t_1}$$

where:

$k$  = thermal conductivity,

$q$  = heat input,

$\theta$  = temperature,

$t$  = time after initiation of heat generation,

$t_1$  = time of initial temperature measurement ( $\theta_1$ ),

$t_2$  = time of final temperature measurement ( $\theta_2$ ).

Thermal conductivity data are presented in Table 30. Conductivity of Saffil Alumina and Fiberchrome either decreased or increased less than 10% (satisfactory performance) as a result of 1000 and 1200°C exposure. Kaowool exhibited a 28 to 29% increase, which can be attributed to its shrinkage in thickness. The low conductivity of Saffil Alumina HT after 100 hours at 1000°C can be attributed to its expansion. After 500 hours at 1400°C, all three blanket materials had undergone a significant conductivity increase. This increase, along with the corresponding density change is summarized below. The density of Saffil Alumina HT decreased while that of Kaowool 1400 and Fiberchrome increased after 500 hours at 1400°C. This would indicate that the conductivity increase is due to a combination of density and crystallographic changes.

<u>Material Designation</u>	<u>Thermal Conductivity</u>	
	<u>Density Change, %</u>	<u>Increase, %</u>
Saffil Alumina HT	-5	65
Kaowool 1400	95	57
Fiberchrome	38	69

## V.

INDIVIDUAL FIBER CAKE AND BLANKET PERFORMANCE SUMMARY

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Based on specimen evaluations and the dimensional and weight loss data generated for fiber cake and blanket samples, performance of the insulation materials are summarized in the following paragraphs.

Irish Refrasil Fiber Cake - Good dimensional stability at 1000°C with little change beyond 25 hours. Thickness increase at 1200°C. Moderate weight loss (4 to 6%). Severe color change reflecting loss of chromia, which retards the devitrification of silica; green color (chromia) completely faded after 350 hours at 1000°C. Material was noncrystalline after 500 hours at 1000°C but exhibited a strong crystobalite peak after 50 and 100 hours at 1200°C. Crystobalite formation has been shown to cause dimensional distortion and cracking in silica RSI tile. Consequently, Refrasil fiber cakes were not tested beyond 1200°C.

Saffil Alumina HT Fiber Cake - Apparent fiber sintering with large shrinkage, yielding a hard RSI-like material with a ceramic ring. Shrinkage was 4 to 6% at 1000°C with little change beyond 25 hours; material was rigid and bowed after 350 hours. Continual shrinkage at 1200°C; material had board-like consistency after 25 hours. Shrinkage was greater than 15% at 1400°C; samples were rigid and bowed after 5 hours. Shrinkage after 25 hours at 1600°C was on the order of 25% and weight loss was about 3%. Large sample-to-sample variations in shrinkage at 1200° and 1400°C. Material readily absorbed vaporized chromia from Irish Refrasil and Fiber-chrome. Retest of a different fiber batch for 25 hours at 1400°C yielded much lower weight loss and shrinkage values, indicating that the poor thermal stability of the old fiber batch was attributable to improperly processed fibers.

Saffil Zirconia HT Fiber Cake - Samples puffed up in thickness and exhibited some delaminations after initial exposures at 1000°C and 1200°C. Changes in weight, length, and width were small. Material was soft and pliable after 500 hours at 1000°C but had become semirigid after 200 hours at 1200°C. Color after 500 hours at 1000° and 1200°C was nearly white. Color changed from off-white to yellow during 1400°C exposure. Samples were semirigid with hard surfaces after 25 hours at 1400°C and board-like after 100 hours. No thickness change occurred at that temperature. Grain growth after 500 hours was extensive. Material was hard and crusty after 5 hours at 1600°C and shrinkage after 25 hours was 10% in length and width and 30% in thickness.

B&W Mullite Fiber Cake - Moderate thickness increases occurred during 1000 and 1200°C runs with 2 to 3% weight loss after 500 hours. Thickness decrease was 6% after 100 hours at 1400°C with 4% weight loss. Length and width changes were slight at these temperatures. Samples were soft and pliable after 500 hours at 1000 and 1200°C but remained flat and square. Samples were semi-flexible with rounded corners after 100 hours at 1400°C and fibers appeared to be brittle. Color remained white at all exposures. Grain growth was noted in most fibers after 500 hours at 1400°C. Fibers were severely embrittled after exposure of 1600°C. Material was semirigid after 25 hours and became increasingly fragile at longer exposures. Shrinkage in thickness was approximately 16% after 25 hours.

3 Micron Mullite Fiber Cake - Material was very fragile after thermal exposure and could not be handled without breaking. The B&W Mullite fiber, by comparison, forms a much sturdier fiber cake. Dimensional and weight changes were similar to B&W Mullite except that thickness decrease was close to 20% after 75 hours at 1400°C. Samples stiffened and became semiflexible after 175 hours at 1400°C and after 25 hours at 1600°C. Color remained white. Severe grain growth and fiber fusion was observed after 200 hours at 1600°C.

Fiberfrax H Fiber Cake - Samples exhibited only slight weight loss at all exposures at 1400°C and changes in length and width were small. Thickness decrease was of the order of 10-20% after 100 hours at 1000 to 1400°C. At 1200°C, thickness decrease was 17% after 100 hours but only 9% after 500 hours. Samples retained their white color and were sturdy and tough after thermal exposure. No significant change in texture was observed although fiber embrittlement and some grain growth was noted after 500 hours at 1400°C. Samples collapsed during a 5-hour exposure at 1600°C and severe grain growth and fiber fusion had occurred.

Fiberfrax H Blanket - Samples were distorted with curled up corners and crusty surfaces after 5 hours at 1000 and 1200°C and were severely deformed and twisted after 100 hours. Because of these deformations, quantitative dimensional changes are largely meaningless and samples were not exposed at 1400 and 1600°C. These deformations are probably significantly influenced by furnace heat-up rates. Weight loss was slight at 1000 and 1200°C and similar to the Fiberfrax H fiber cake samples. Original fibers were noncrystalline but moderately strong mullite peaks were observed after 500 hours at 1000°C and after 50 hours at 1200°C. The blanket material had been purchased in December 1972 and therefore represents an early lot of Fiberfrax H production blanket. Evaluation of this material by Carborundum indicated that it met specifications with respect to fiber diameter and fiber chemistry (ref. 10).

Kaowool Blanket - Samples exhibited only slight weight loss at all test temperatures to 1400°C and changes in length and width were small. Thickness had decreased 20% after 25 hours at 1000°C and 1200°C, but no further decrease occurred between 25 and 500 hours. Thickness decrease at 1400°C was 25% after 25 hours and 40% after 100 hours. Specimens were flexible before exposure and gradually became semiflexible at the test temperature. Their original white color was retained. Samples remained flat and square at 1000°C. Edges and corners started to curl up after 100 hours at 1200°C and after 50 hours at 1400°C. For 1000 and 1200°C applications, thickness can be stabilized by temperature exposure before use. Severe shrinkage and sample collapse had occurred after 25 hours at 1600°C, accompanied by severe grain growth and fiber fusion.

Fiberchrome Blanket - Samples lost chromia as denoted by color changes from gray to pale green-gray. Weight loss was 4% after 25 hours at temperatures to 1400°C and 6% after 500 hours at 1000 and 1200°C. The material was very stable dimensionally to 500 hours at 1000 and 1200°C, and to 100 hours at 1400°C. A 10% shrinkage in thickness was noted after 500 hours at 1200°C and after 100 hours at 1400°C. Samples remained flat and square after exposures to 1400°C. They were soft and drapable after 500 hours at 1000°C but became semirigid at 1200 and 1400°C. Extensive grain growth with fiber fusion was noted after 500 hours at 1400°C and fibers were strongly crystalline with pronounced crystobalite and mullite peaks. Exposure at 1600°C resulted in severe shrinkage and sample collapse after 25 hours, accompanied by fiber fusion.

Saffil Alumina HT Blanket - Blanket contains an organic binder that oxidizes during initial temperature exposure, yielding a weight loss of 8 to 10%. Blanket is made up of two distinct layers and puffed up in thickness during thermal exposure. Thickness increase was 50% at 1000 and 1200°C, and 20% at 1400°C. Length and width remained unchanged at 1000°C and decreased 4 to 6% after 500 hours at 1200°C and 2 to 4% after 500 hours at 1400°C. Blanket remained soft and flexible after exposures to 1400°C. Samples took on a pink color during exposure due to chromia contamination from Fiberchrome. Material exhibited strong alpha alumina peaks after 500 hours at 1400°C. Samples developed a semihard crust after 5 hours at 1600°C and were rigid with curled up corners after 100 hours. Shrinkage after 25 hours at 1600°C was 5% in length, 12% in width, and 22% in thickness. Severe fiber sintering and grain growth was observed.

Saffil Zirconia HT Blanket - Blanket contains an organic binder that oxidizes during initial temperature exposure, yielding a weight loss of 6 to 8%. Blanket is made up of two distinct layers. It remained soft and flexible during 1000°C exposure; thickness puffed up 50% and length and width decreased 0 to 2%. The material became semiflexible at 1200°C and edges began to curl up after 100 hours. There was no thickness change after 25 hours and a 26% thickness decrease after 425 hours. At 1400°C, the material became semirigid; edges began to curl up after 5 hours, and a hard crusty surface was noted after 25 hours. Shrinkage at 1400°C was severe. Thickness had decreased 50% and length and width 12 to 14% after 100 hours. Shrinkage in length and width was 20% after 500 hours. Samples were hard and crusty after 5 hours at 1600°C. Sample collapse, accompanied by severe grain growth and fiber sintering occurred after 25 hours at 1600°C.

Microquartz Blanket - Material hardened and surface became crusty during 1000 and 1200°C exposures. Specimens were bowed and had uneven surfaces. Large shrinkage occurred, especially in thickness. Shrinkage was more severe in the gas kiln than in the electric kiln samples. Because of unsatisfactory dimensional stability, samples were not tested at 1400 and 1600°C. According to Johns-Manville, the observed shrinkage is natural for Microquartz blanket because of the high purity and porosity of the fibers. Shrinkage increases with decreasing impurity level while the tendency to devitrify decreases. A high temperature heat treatment of the fibers would eliminate the observed shrinkage and weight loss (ref. 11).

Based on the satisfactory performance criteria of linear shrinkage (less than 15%), density increase (less than 30%), and room temperature thermal conductivity (less than 10% increase for blankets), the following materials were rated satisfactory for 500 hours exposure at 1000, 1200 and 1400°C. None of the materials could be rated satisfactory after 25 hours at 1600°C. The effects of crystallographic changes must be investigated in more detail before performance criteria can be established.

500 hr at 1000°C

500 hr at 1200°C

500 hr at 1400°C

Fiber Cakes

Irish Refrasil

Irish Refrasil

Saffil Alumina HT  
(new fiber lot)

Saffil Alumina HT  
(new fiber lot)

Saffil Alumina HT  
(new fiber lot)

B&W Mullite

Saffil Zirconia HT

B&W Mullite

B&W Mullite

3 Micron Mullite

3 Micron Mullite

Fiberfrax H

Blankets

Fiberchrome

Fiberchrome

Saffil Alumina HT

Saffil Alumina HT

Saffil Zirconia HT

It is evident that more fiber cakes than blankets met the shrinkage and density change criteria after elevated temperature exposure in the 1000 to 1400°C range. The Saffil Alumina HT blanket had satisfactory performance for shrinkage and density change, but failed to meet the thermal conductivity criterion after 500 hours at 1400°C. Dimensional instability in blankets were probably caused by the preferred orientation of the fibers and by the burn-off and chemical reactivity of organic binders and other processing aids used in the fabrication of blanket insulation.

The three fiber materials chosen for Task II RSI (Reusable Surface Insulation) fabrication were Saffil Alumina HT, Saffil Zirconia HT, and B&W Mullite.

B&W Mullite was selected because of its high dimensional stability to 1400°C and its adequate performance for short time (5 hours) at 1600°C. In addition, mullite provides a suitable comparison standard for other fibers since mullite systems had been extensively investigated under Contract NAS1-10533 (Refs. 12 and 13).

The Saffil Alumina HT and Zirconia HT fibers were selected for the following reasons:

- 1) The fiber systems are new and have not been previously investigated;
- 2) The fibers are low cost--\$20.95 to 25.35/kg (\$9.50 to 11.50/lb);
- 3) The fibers are of uniform diameter and free of shot content;
- 4) Zirconia exhibited no grain growth and only slight increase in crystallinity after 500 hours at 1200°C;
- 5) Zirconia exhibited a low weight loss and good dimensional stability after 100 hours at 1400°C;
- 6) Alumina (new fiber lot) exhibited a low weight loss and good dimensional stability after 25 hours at 1400°C;
- 7) Alumina fibers (blanket material) exhibited only slight evidence of grain growth and some increase in crystallinity after 500 hours at 1200°C;
- 8) Fibercakes of 0.11 to 0.12 g/cc (6.9 to 7.5 lb/ft<sup>3</sup>) density could be readily produced.

RSI processing studies were conducted with the following binder systems, using binder solids-to-fiber ratios of 1:5, 1:10, and 1:20:

Colloidal Silica	Nalco E-136, Nalco Chemical Company Ludox AS, E. I. DuPont DeNemours and Company
Colloidal Alumina	
Ethyl Silicate	Silbond H-4, Stauffer Chemical Company
Aluminum Phosphate	Winnofos, ICI Mond Division
Colloidal Silica & Alumina	(28.1% SiO <sub>2</sub> ; 71.9% Al <sub>2</sub> O <sub>3</sub> )
Colloidal Alumina & Ethyl Silicate	(28.1% SiO <sub>2</sub> ; 71.9% Al <sub>2</sub> O <sub>3</sub> )

Three techniques for incorporating binders into the fiber cakes were utilized:

- 1) The fiber cakes were water felted with Separan AP-30 deflocculating agent, oven dried, and reimpregnated with the binder solution. To retain the binder, the solution was either gelled *in situ* by pH adjustment or a cationic starch (CATO-75, National Starch and Chemical Corporation) was added to promote binder adsorption on the fiber surface.
- 2) Binder and cationic starch were added directly to the felting water.
- 3) RSI tiles containing Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> binders in combination were also prepared by adding Alon and cationic starch to the felting water. Following oven drying, the fiber cakes were reimpregnated with colloidal silica or Silbond H-4 ethyl silicate.

Impregnation with ethyl silicate (1:5 SiO<sub>2</sub> binder-to-fiber ratio) was accomplished by preparing an alcohol solution of 14.2 parts by volume Silbond H-4 and 71.1 parts by volume isopropyl alcohol. Addition of 13.9 parts by volume distilled water and 0.8 parts by volume concentrated NH<sub>4</sub>OH caused gelling to occur within 2-1/2 minutes.

To impregnate with colloidal silica, Nalco E-136 or Ludox AS was diluted with distilled water to obtain the desired binder (SiO<sub>2</sub> solids)-to-fiber ratio. The volume of binder solution used for impregnation was always equal to the nominal volume of the fiber cake. Ludox AS was gelled by HCl addition to adjust the water solution to pH 5.5. Colloidal alumina (Alon) was gelled by NH<sub>4</sub>OH addition to adjust the water solution to pH 9.1. Combinations of colloidal alumina and silica contained 28.1% by weight

$\text{SiO}_2$  solids and 71.9% by weight  $\text{Al}_2\text{O}_3$  solids. This is the chemical equivalent of mullite,  $3 \text{Al}_2\text{O}_3 \times 2 \text{SiO}_2$ .

To facilitate binder solids retention through the use of cationic starch, a 3% solution of starch in water was first prepared. The proper amount of binder was then added to the starch solution and either used directly as the felting liquid (Winnofos aluminum phosphate impregnation) or as the secondary impregnant for fiber cakes (Nalco E-136 colloidal silica impregnation).

All RSI felts were fired for 30 minutes at  $1371^\circ\text{C}$  ( $2500^\circ\text{F}$ ). The amount of binder retained in the fired RSI tiles was less than the amount indicated by the binder-to-fiber ratio since some binder solids were lost with the felting or impregnation liquid.

Twelve RSI materials involving combinations of the three fiber systems, six binders, and three binder-to-fiber ratios were prepared as 12.7 cm dia  $\times$  1.9 cm (5 in. dia.  $\times$  3/4 in.) tile and screened by furnace exposure for 22 hours at  $1200^\circ\text{C}$  ( $2192^\circ\text{F}$ ) plus 5 hours at  $1400^\circ\text{C}$  ( $2552^\circ\text{F}$ ). Resultant weight and dimensional changes were measured and flexural properties of exposed specimens and unexposed controls were determined. Based on these evaluations, Silbond H-4 at a binder-to-fiber ratio of 1:5 was selected as the impregnant for additional tile characterizations. Specifically, Silbond H-4 was chosen for the following reasons:

- 1) Silbond-impregnated tile samples exhibited excellent dimensional and weight stability after  $1200^\circ\text{C}$  and  $1400^\circ\text{C}$  exposures;
- 2) Flexural strengths were high and did not decrease significantly as a result of high temperature exposures;
- 3) Elongations were as high or higher than for other impregnants;
- 4) Impregnation and gellation of Silbond allowed binder pickup to be controlled with a good degree of precision;
- 5) Impregnation appeared to be uniform through the thickness of tile samples.

Twenty percent by weight binder solids was selected since it yielded a sturdier tile than lower binder contents. Gellation and binder pickup of Nalco E-136 and Ludox AS was more difficult to control than for Silbond H-4. This may account for the less satisfactory results obtained with these systems. Combinations of colloidal alumina and silica yielded inconsistent results.

However, only one alumina-to-silica ratio was tried and it is possible that a mixed binder with a lower alumina-to-silica ratio would yield a high strength system that is more refractory than silica binders.

Ten tile pieces in sizes of 12.7 cm dia x 3.2 cm (5 in. diam x 1-1/4 in.), 15.2x6.4x2.5 cm (6x2-1/2x1 in.), and 15.2x6.4x3.8 cm (6x2-1/2x1-1/2 in.) were prepared from each of the three fibers. Densities averaged 0.150 g/cc (9.4 lb/ft<sup>3</sup>) for alumina tiles, 0.177 g/cc (11.0 lb/ft<sup>3</sup>) for zirconia tile, and 0.156 g/cc (9.7 lb/ft<sup>3</sup>) for mullite tile. The higher density of the Zirconia RSI is caused by a reduction in fiber cake thickness during drying after felting and impregnation. Binder pickup averaged 14.6% of fiber weight for alumina, 12.9% for zirconia, and 14.5% for mullite. Linear firing shrinkage for all tile materials and configurations was less than 2-1/2%.

The 15.2x6.4x2.5 cm tiles were used as density/shrinkage samples after light sanding to remove surface irregularities. Flexural specimens, 15.2x1.9x1.3 cm (6x3/4x1/2 in.) in size and SEM/XRD specimens, 5.1x1.9x1.3 cm (2x3/4x1/2 in.) in size were cut from the 15.2x6.4x3.8 cm tile with a diamond-plated wheel. Thermal conductivity specimens, 10.2x5.1x2.5 cm (4x2x1 in.) in size with chamfered corners, were cut from the 12.7 cm dia x 3.2 cm tile. Density/shrinkage samples were weighed and measured. Control samples for flexural, thermal conductivity, and SEM/XRD measurements were set aside. The remaining samples were divided into two groups for exposure to 25 thermal/vacuum cycles at 1200°C and at 1400°C.

For thermal cycling, all specimens were placed on fibrous insulation so that their upper surfaces were at the same level. Two 30.5x30.5 cm (12x12 in.) water-cooled quartz lamp reflectors were used for heating. The quartz lamp array and specimens were placed in a Tenney Vacuum Chamber (Model 4D5) with inside dimensions of 122 cm dia x 152 cm (48 in. dia x 60 in.). A thermal cycle consisted of a 600-sec heating period, a 400-sec hold at peak temperature, and a cooldown period. Tile samples instrumented with Pt-6% Rh vs Pt-30% Rh surface thermocouples were used to monitor and control tile surface temperatures. A typical 1200°C heat cycle is shown in Fig. 57. Pressure during the 400-sec hold at 1200°C was approximately 10 torr or 29.3 km (96,000 ft) equivalent altitude. Arcing from the quartz lamp electrical connections to the specimen surfaces occurred at low pressure during the initial 1400°C runs. To prevent arcing, pressure was increased to approximately 32 torr or 21.6 km (71,000 ft) equivalent altitude. The 1200°C samples retained their original color, but the arcing caused some surface discoloration in the 1400°C samples.

## VII. RSI TEST RESULTS

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### A. WEIGHT, DIMENSIONAL AND DENSITY CHANGES

Weight loss, dimensional changes, and density changes resulting from the 25 radiant heat cycles at 1200 and 1400°C were minimal as shown in Table 31. Average weight loss was less than 0.5%, average dimensional change was less than 3.5%, and average density change was less than 3%. No bowing or other deformation was noted in the exposed samples.

### B. SCANNING ELECTRON MICROSCOPY

No changes in fiber morphology due to radiant heating at 1200 and 1400°C were detected by comparing electron micrographs of exposed samples and unexposed controls. Micrographs at 300X magnification (controls and 1400°C exposure) are shown in Figures 58, 59, and 60.

### C. X-RAY DIFFRACTION

RSI samples exposed to 25 thermal/vacuum cycles and unexposed controls were subjected to X-Ray diffraction analyses. The Saffil Alumina controls exhibited moderate crystallinity, comprising a mixture of alpha alumina (corundum), alpha cristobalite ( $SiO_2$ ), and traces of mullite. Samples exposed to surface temperatures of 1200° and 1400°C exhibited the same peaks and no increase in peak intensity due to thermal exposure was noted.

The Saffil Zirconia controls were strongly crystalline. The crystal form was tetragonal  $ZrO_2$ , and a trace of alpha cristobalite ( $SiO_2$ ) was observed. Crystal structure and peak intensity did not change with thermal/vacuum exposure.

The B&W mullite controls exhibited moderate crystallinity, comprising mullite peaks. There was no evidence of alpha cristobalite. Samples exposed to surface temperatures of 1200°C and 1400°C exhibited the same peaks and similar peak intensities as control samples.

#### D. FLEXURAL PROPERTIES

Room temperature flexural strengths, moduli of elasticity, and strains at failure of RSI specimens are listed in Table 32. Listed values are the average of three tests. Specimens were tested in four-point bending as beams of 13.35 cm (5.25 in.) span and 4.45 cm (1.75 in.) between load points. Load and support points were 0.64 cm (1/4 in.) radii. Flexural properties were not degraded by the 25 thermal/vacuum cycles; for Saffil Zirconia RSI, flexural properties actually appear to improve with exposure temperature. Flexural strengths for Alumina and Zirconia RSI averaged approximately  $500 \times 10^3$  N/m<sup>2</sup> (72 psi) and this is an acceptable value for a 0.16 g/cc (10 lb/ft<sup>3</sup>) density material. In contrast, the Mullite RSI strength of  $215 \times 10^3$  N/m<sup>2</sup> (31 psi) was less than half that of alumina and zirconia, its modulus of elasticity was only 1/5 that of alumina and zirconia, but its strain at failure was twice that of the alumina and zirconia specimens.

#### E. ROOM TEMPERATURE THERMAL CONDUCTIVITY

Room temperature thermal conductivity of RSI specimens was measured by the line source technique as described in Chapter IV. Results are summarized in Table 33. The type of fiber had a significant influence on conductivity with Alumina RSI exhibiting the highest conductivity of 0.170 W/m-°K (1.18 Btu-in./hr-ft<sup>2</sup>-°F) and Zirconia RSI exhibiting the lowest conductivity of 0.052 W/m-°K (0.36 Btu-in./hr-ft<sup>2</sup>-°F). Mullite RSI had a room temperature conductivity of 0.069 W/m-°K (0.48 Btu-in./hr-ft<sup>2</sup>-°F) for the unexposed controls. The thermal conductivity of Zirconia RSI did not change as a result of thermal/vacuum cycling while the conductivity of Alumina and Mullite RSI decreased between 13 and 20% as a result of the cyclic exposures.

#### F. INDIVIDUAL RSI PERFORMANCE SUMMARY

Based on thermal/vacuum exposures for 25 cycles to surface temperatures of 1200°C and 1400°C, and on subsequent evaluations, the performance of the individual RSI materials is summarized in the following paragraphs.

*Saffil Alumina HT RSI* - Impregnation with ethyl silicate was readily controlled, yielding RSI tile of 0.15 g/cc (9.3 lb/ft<sup>3</sup>) density. No significant specimen deformation, weight loss,

dimensional shrinkage, or change in fiber morphology resulted from the thermal/vacuum exposures. Flexural properties were unaffected while room temperature thermal conductivity decreased approximately 19% as a result of the 25 exposure cycles to 1200°C and to 1400°C.

*Sj'ill Zirconia HT RSI* - Zirconia fiber cakes tended to shrink in thickness during oven drying after felting and after ethyl silicate impregnation, resulting in RSI tile of 0.18 g/cc (11 lb/ft<sup>3</sup>) density. Binder pickup of 12.9% based on fiber weight was less than for Alumina or Mullite RSI, possible because the higher density of zirconia fibers resulted in a lower fiber volume in the zirconia fiber cake for equivalent fiber cake density. No significant specimen deformation, weight loss, dimensional shrinkage, or change in fiber morphology resulted from the thermal/vacuum exposures. Flexural properties tended to increase as a result of 25 thermal exposure cycles while room temperature thermal conductivity was unaffected.

*B&W Mullite RSI* - Impregnation with ethyl silicate was readily controlled, yielding RSI tile of 0.16 g/cc (9.7 lb/ft<sup>3</sup>) density. Flexural strength was only 40% of the comparative strength of Alumina RSI, modulus of elasticity was approximately 1/4 that of Alumina RSI, while strain at failure was twice that of Alumina RSI. Room temperature thermal conductivity was 33% higher than that of Zirconia RSI. No significant specimen deformation, weight loss, dimensional shrinkage, or change in fiber morphology resulted from the thermal/vacuum exposures. Flexural properties were unaffected while room temperature thermal conductivity decreased approximately 15% as a result of 25 exposure cycles to 1200°C and to 1400°C.

## VIII. FABRICATION COST ANALYSIS

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Material and fabrication costs for 930 m<sup>2</sup> (10,000 ft<sup>2</sup>) of blanket materials and of RSI tile were generated. Blanket insulation costs involve direct material cost only since cutting, fitting, and attachment would have to be specified by engineering drawings and priced in accordance with the complexity of these operations. Material costs for three blanket insulations in quantity of 930 m<sup>2</sup> (10,000 ft<sup>2</sup>) are as follows:

Kaowool 1400; 1.27 cm (0.50 in.) thick; 0.13 g/cc (8 lb/ft <sup>3</sup> ) density	\$12,600
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Fiberchrome; 1.27 cm (0.50 in.) thick; 0.13 g/cc (8 lb/ft <sup>3</sup> ) density	\$12,800
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Saffil Alumina HT; 1.90 cm (0.75 in.) thick; 0.08 g/cc (5 lb/ft <sup>3</sup> ) density	\$38,400
---	----------

Fabrication costs for 930 m<sup>2</sup> (10,000 ft<sup>2</sup>) of 2.5 cm (1 in.) thick RSI tile are based on Saffil Alumina HT fiber and ethyl silicate (Stauffer Silbond H-4) binder. The RSI would be made as blocks 43.2x43.2x6.4 cm (17x17x2-1/2 in.) in size and eight individual tiles, 20.3x20.3x2.5 cm (8x8x1 in.) would be cut from each block. The 3000 blocks (24,000 tiles) would yield 990 m<sup>2</sup> (10,650 ft<sup>2</sup>) of RSI and allow a scrappage factor of 6.5% to produce the required amount. Raw materials requirements for this quantity of RSI are as follows:

Saffil Alumina HT Fiber	4500 kg (10,000 lb)
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Silbond H-4	5000 kg (11,000 lb)
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Isopropyl Alcohol	21,000 kg (47,000 lb)
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Concentrated NH <sub>4</sub> OH	300 kg (650 lb)
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Separan AP-30	80 kg (170 lb)
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To produce the designated quantity of RSI tile over a time period of one year requires a group of eight people consisting of:

6 Manufacturing Technicians

1 Supervisor

1 Quality Control Inspector

Man-hours allocated to the various operations involved in RSI production are as follows:

Material preparation	2000 hr
Felting	4000 hr
Drying	500 hr
Impregnation	4000 hr
Firing	500 hr
Machining	1000 hr
Supervision	2000 hr
Inspection	2000 hr
Shipping	384 hr
Contract support	200 hr

Total material and fabrication cost for 930 m<sup>2</sup> (10,000 ft<sup>2</sup>) RSI tile is \$513,130, based on current material and labor costs. The cost breakdown is summarized in Table 33. The corresponding unit cost for an individual RSI tile, 20.3x20.3x2.5 cm (8x8x1 in.) in size is \$21.38.

## IX. SUMMARY OF RESULTS

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### A. FIBER CAKES

Irish Refrasil, Saffil Alumina HT, and Saffil Zirconia HT fibers had a narrow fiber diameter range and were essentially free of shot content.

Separan AP 30 deflocculating agent, added to the felting water in a concentration of 0.05% was found to be very beneficial to producing water-felted fiber cakes. A 0.12 g/cc fiber cake density could be readily attained with Irish Refrasil, Saffil Alumina HT, Saffil Zirconia HT, and B&W Mullite fibers. Fiberfrax H and 3 Micron Mullite shrunk during oven drying and yielded higher fiber cake densities. Three Micron Mullite fiber cakes were fragile and broke during handling after furnace exposure.

A density increase greater than 30% or a linear shrinkage greater than 15% resulting from thermal exposure denotes unsatisfactory performance. On that basis, all fiber cakes were rated satisfactory for 500 hours at 1000°C; only Saffil Alumina HT (new fiber lot) and B&W Mullite were rated satisfactory for 500 hours at 1400°C and none of the fiber cakes were satisfactory for even 25 hours at 1600°C. B&W Mullite was the only fiber cake that did not become hard and crusty or experience large shrinkage or sample collapse after 5 hours at 1600°C.

Crystallinity of fiber cakes increased with exposure temperature. Irish Refrasil remained noncrystalline after 500 hours at 1000°C while the strongly crystalline structure of Saffil Zirconia HT was unchanged by the 1000°C exposure. All other fiber cakes exhibited weak crystalline peaks after 500 hours at 1000°C.

No change in fiber grain size occurred as a result of 1000°C exposure. 1200°C exposure produced grain growth in Saffil Alumina HT (old material lot) while other fiber cakes were unchanged, based on SEM observations. All fiber cakes exhibited grain growth after 500 hours at 1400°C.

### B. BLANKET MATERIALS

Saffil Alumina HT and Fiberchrome were the two best blanket insulations, based on dimensional stability, performing satisfactory at 1400°C for 500 and 100 hours respectively. The length, width

and thickness of Saffil Alumina HT shrunk less than 6% at 1400°C. Kaowool 1400 exhibited the least grain growth and the lowest weight loss after 500 hours at 1400°C. Shrinkage of Kaowool 1400 after 500 hours at 1200°C and after 100 hours at 1400°C was less than 6% in length and width but more than 20% in thickness.

Fiberfrax H and Microquartz blanket became rigid and deformed during furnace exposure at 1000 and 1200°C and dimensional changes were therefore difficult to measure. Saffil Alumina and Zirconia HT blankets contained an organic binder that burned off during initial furnace exposures and resulted in a weight loss of 10% and 7% respectively.

No change in grain size occurred as a result of 1000°C exposure. 1200°C exposure produced slight grain growth in Saffil Alumina HT blanket and evidence of fiber embrittlement in Fiberfrax H blanket. All blankets exhibited grain growth after 500 hours at 1400°C.

Room temperature thermal conductivity of Saffil Alumina HT blanket was approximately 20% higher than that of Fiberchrome and Kaowool before thermal exposure. Thermal conductivity of Saffil Alumina HT and Fiberchrome increased less than 10% (acceptable performance) as a result of 1000 and 1200°C exposure for 500 hours while that of Kaowool 1400 increased approximately 28%. The three blanket materials exhibited thermal conductivity increases greater than 50% after 500 hours at 1400°C.

#### C. RSI MATERIALS

Uniform Reusable Surface Insulation (RSI) was produced by impregnating water-felted and dried fiber cakes with ethyl silicate and gelling the binder in situ. Using binder solids in an amount of 20% of fiber weight in the impregnation solution, binder pickup after firing ranged from 12.9 to 14.6% of fiber weight. RSI tile densities of 0.150 and 0.156 g/cc could be repeatably achieved with Saffil Alumina HT and B&W Mullite fibers. Using identical techniques, Saffil Zirconia HT fibers yielded tile densities of 0.177 g/cc. Firing RSI tile for 30 minutes at 1371°C (2500°F) produced tile of good strength and thermal stability. Linear firing shrinkage was less than 2%.

Exposure to 25 thermal/vacuum cycles at surface temperatures of 1200 and 1400°C did not significantly change the weight, size, density, and fiber morphology, nor degrade strength, elongation, or thermal conductivity of the RSI tile specimens.

Flexural strengths of Saffil Alumina HT and Saffil Zirconia HT RSI were more than twice and their moduli of elasticity were nearly five times that of B&W Mullite RSI. Percent strain at failure of Saffil Alumina and Zirconia HT was approximately 50% of the failure strain of B&W Mullite.

Room temperature thermal conductivity of Saffil Alumina HT RSI was approximately three times higher than that of Saffil Zirconia HT RSI and  $2\frac{1}{2}$  times higher than that of B&W Mullite RSI before exposure. After exposure, it was reduced to a little over twice.

#### D. FABRICATION COST ANALYSIS

Material and fabrication costs for  $930\text{ m}^2$  ( $10,000\text{ ft}^2$ ) of blanket materials and of RSI tile were generated.

Fibrous insulation materials can be considered low cost for aerospace application if their price is less than \$30 per kg. The cost of two materials included in this study, Mullite fibers and Irish Refrasil fibers exceeded that amount.

The cost of Fiberchrome and Kaowool 1400 blanket in production quantity is approximately 1/3 that of Saffil Alumina HT blanket.

Based on current material and labor costs, it would cost \$513,000 to make  $930\text{ m}^2$  ( $10,000\text{ ft}^2$ ) of RSI tile (8x8x1 in.) using Saffil Alumina HT fiber and the preparation technique developed in the program. Cost is broken down as 60% labor, 30% raw material, and 10% profit.

CONCLUSIONS AND RECOMMENDATIONS

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## CONCLUSIONS

Time-temperature exposures of fiber cakes, blankets, and RSI tiles revealed that RSI tiles are less susceptible to weight, dimensional and crystallographic changes than fiber cakes or blankets. This can be attributed to the stabilizing effects of the 1371°C (2500°F) RSI firing cycle and to the use of a rigid binder which locks the fibers in place. After 25 hours at 1400°C, the average density change in fiber cakes ranged from 1.8% decrease (B&W Mullite) to 43% increase (Saffil Alumina HT-old fiber lot) and the average density change in blankets ranged from 19.8% increase (Saffil Alumina HT) to 97.5% increase (Saffil Zirconia HT). In contrast, 25 heat cycles to 1400°C caused average density changes in RSI tile ranging from 0% (Saffil Zirconia HT) to 1.3% (B&W Mullite). Although cumulative RSI tile exposure was only 2.8 hours, comparison of RSI stability with that of fiber cakes and blankets after 25 hours is valid since the observed changes appear to occur early during the exposure period. The greater dimensional and density changes in blankets compared to fiber cakes is probably due to the preferred orientation of fibers and to the use of organic binders and possibly other processing aids in the blanket insulations.

Based on dimensional and density changes and on observed visual appearance after thermal exposure, Saffil Alumina HT (new fiber lot), B&W Mullite and Saffil Zirconia HT were the best fiber cake materials while Saffil Alumina HT and Fiberchrome were the best blanket insulations.

The room temperature thermal conductivity of Saffil Alumina HT blanket decreased 3% as a result of 500 hours exposure at 1200°C, that of Fiberchrome increased 4%, and that of Kaowool 1400 increased 28%. After 500 hours exposure at 1400°C, thermal conductivity had increased 65% for Saffil Alumina HT, 69% for Fiberchrome, and 57% for Kaowool 1400. The changes in room temperature thermal conductivity of RSI tile after thermal cycling at 1200 and 1400°C ranged from 19% decrease to 6% increase.

The flexural strength, strain to failure and modulus of elasticity of Saffil Alumina HT and B&W Mullite RSI was essentially unaffected by thermal cycling at 1200 and 1400°C except for a 13% decrease in failure strain of B&W Mullite after 1400°C cycling. The flexural strength, strain to failure and modulus of elasticity of Saffil Zirconia HT RSI increased 37%, 12%, and 12% respectively after 1400°C cycling.

All materials exhibited grain growth after 500 hours at 1400°C; thus none of the materials tested have true long time stability at that temperature. Crystallographic changes started in some materials at 1000°C. Crystallinity of all materials increased with exposure temperature and became significant between 1200 and 1400°C.

A short time, high temperature exposure of blanket insulations would tend to stabilize their size, weight, density, crystallinity, and grain size before application and increase their potential use temperature.

A qualitative rating base for the materials studied in this program is as follows:

#### BEST FIBER

Low Cost	Fiberfrax H
Uniformity	Irish Refrasil
Weight Loss	Saffil Zirconia HT
Dimensional Stability	B&W Mullite
Chemical Stability	B&W Mullite

#### BEST BLANKET

Low Cost	Fiberchrome
Weight Loss	Kaowool 1400
Dimensional Stability	Saffil Alumina HT
Chemical Stability	Kaowool 1400
Thermal Conductivity	Fiberchrome

#### BEST RSI

Low Cost	Saffil Alumina HT
Ease of Processing	Saffil Alumina HT, B&W Mullite
Strength	Saffil Alumina and Zirconia HT
Thermal Conductivity	Saffil Zirconia HT
Thermal Stability	Alumina, Zirconia, Mullite

#### B. RECOMMENDATIONS

The work accomplished has suggested a number of additional investigations which were beyond the scope of the current program. However, these investigations would have to be oriented towards specific applications since it has been shown that oxide fiber insulations have potential for long time aerospace use at 1200°C and could be stabilized to meet shorter duration 1400°C requirements. Investigations would involve evaluation of new oxide fiber systems,

preconditioning of fibers and blankets, studies of RSI tile density effects and optimization of the RSI tile fabrication process to assure quality products capable of meeting the requirements of specified applications.

1. Saffil Fibers as High Temperature Materials. Imperial Chemical Industries Limited, Mond Division.
2. Johns-Manville Aerospace Products, J-M Micro-Quartz Felt. Johns-Manville Aerospace Products.
3. B&W Kaowool 1400 Blanket Properties. Babcock and Wilcox Company, Refractories Division, 1973.
4. B&W Mullite Fiber Properties. Babcock and Wilcox Company.
5. Fiberfrax Product Specifications, Product Data - Fiberfrax H Blanket and Felt. The Carborundum Company, Refractories and Electronics Division, Niagara Falls, NY.
6. Thermal Insulation Products, J-M Fiberchrome Refractory Fiber Felt. Johns-Manville, Denver, Colorado, 1973.
7. Procedure for Dispersing Fiber Bundles. NASA Tech Brief B73-10438, Langley Research Center. Technology Utilization Office, NASA, Washington, D.C., 1973.
8. Separan Polymers Technical Data - Separan AP-30. The Dow Chemical Company, Designed Products Department, Midland, Michigan, 1971.
9. Greenwood, L. R. and Podlaseck, S. E.: Vacuum Induced Effects on Ablative Heat Shield Materials. M-68-20, Martin Marietta Corporation, Denver Division, September 1968.
10. Private communication from Mr. L. Sweet, The Carborundum Company, Niagara Falls, New York, July 1975.
11. Private communication from W. C. Miiller, Johns-Manville, Denver, Colo, December 1974.
12. Tanzilli, R. A.: Development of an External Ceramic Insulation for the Space Shuttle Orbiter. NASA CR-112038, April 1972.
13. Tanzilli, R. A.: Development of an External Ceramic Insulation for the Space Shuttle Orbiter; Part 2 - Optimization, NASA CR-112257, March 1973.

APPENDIX A

FIGURE

- 37

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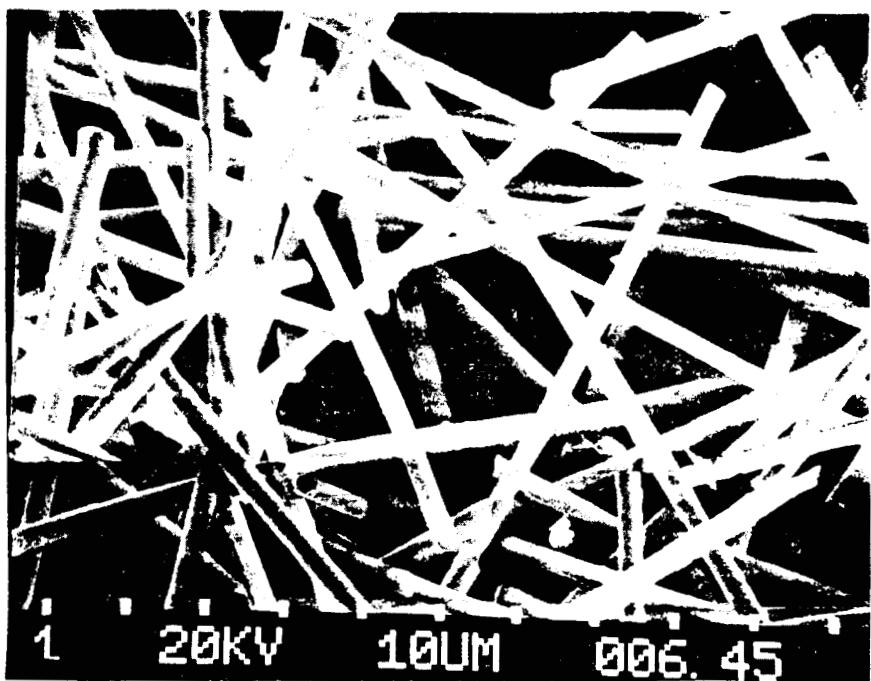


Figure 1.- SEM photograph (1150X) of Irish Refrasil fibers.



Figure 2.- SEM photograph (330X) of Fiberfrax H fibers showing shot content.

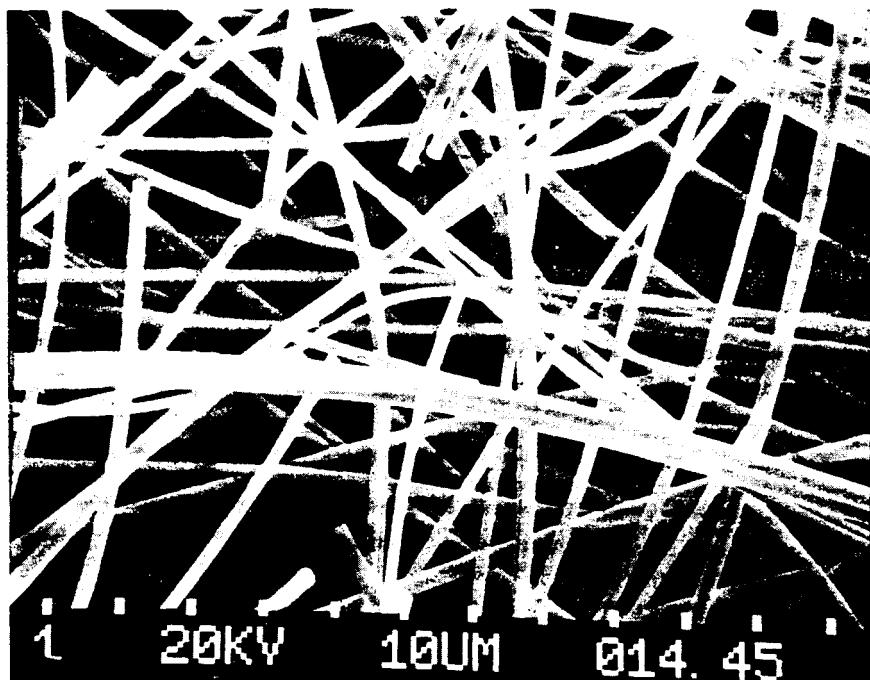


Figure 3.- SEM photograph (1040X) of Saffil Alumina HT fibers.

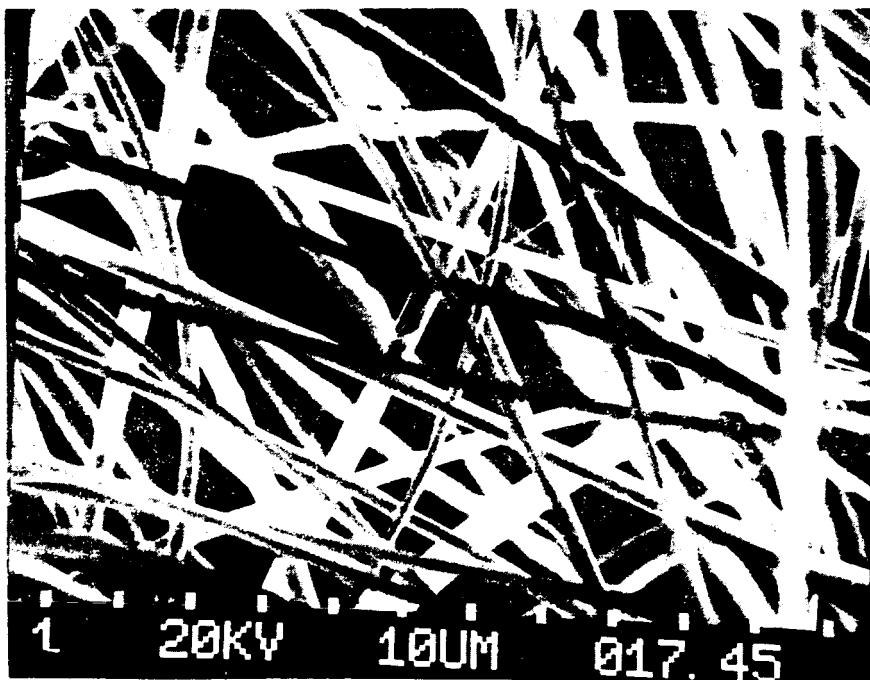


Figure 4.- SEM photograph (1040X) of Saffil Zirconia HT fibers.

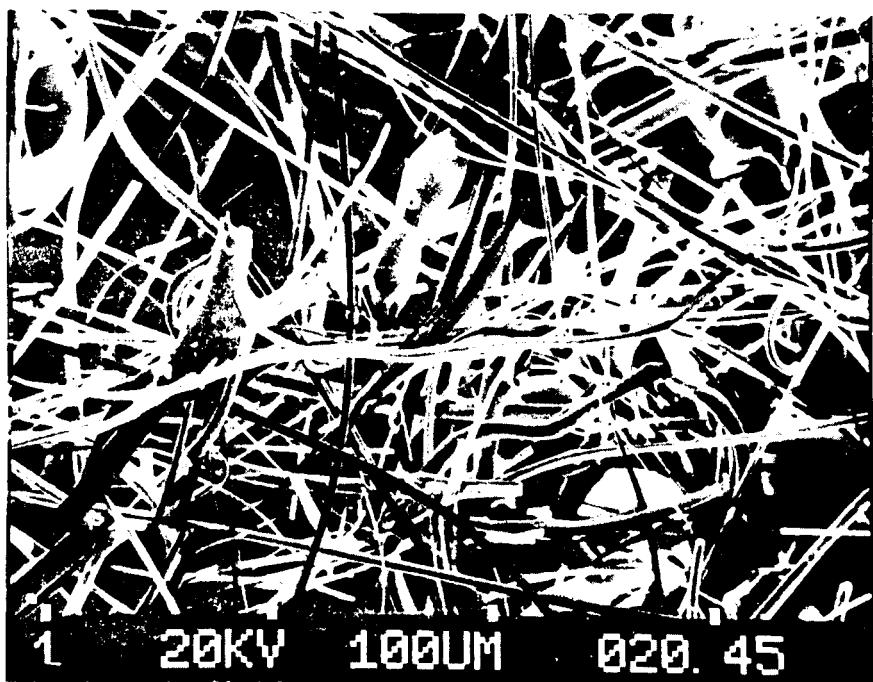


Figure 5.- SEM photograph (320X) of B&W Mullite fibers showing shot content.

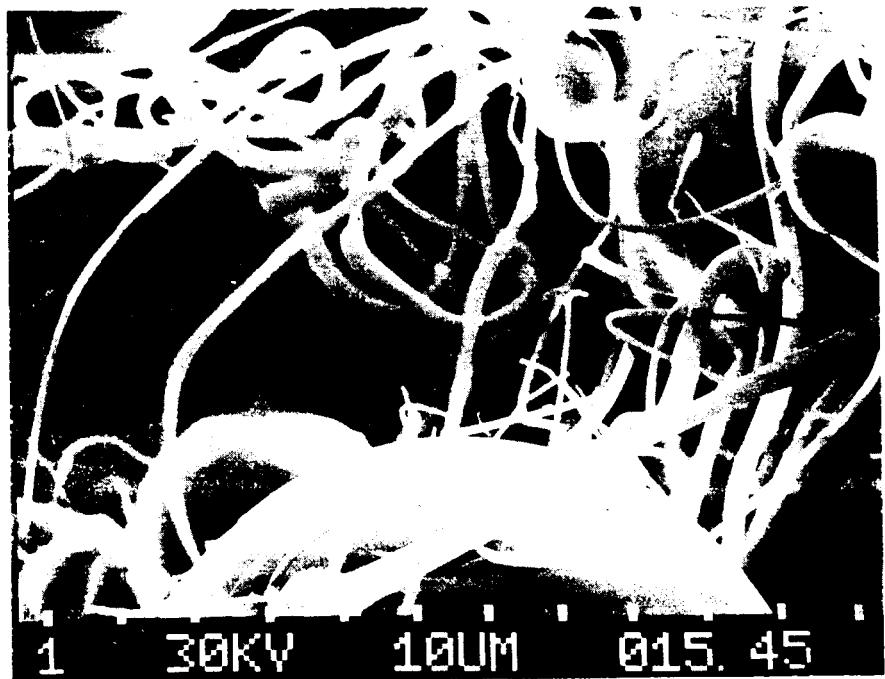


Figure 6.- SEM photograph (1040X) of 3 Micron Mullite fibers showing shot content.

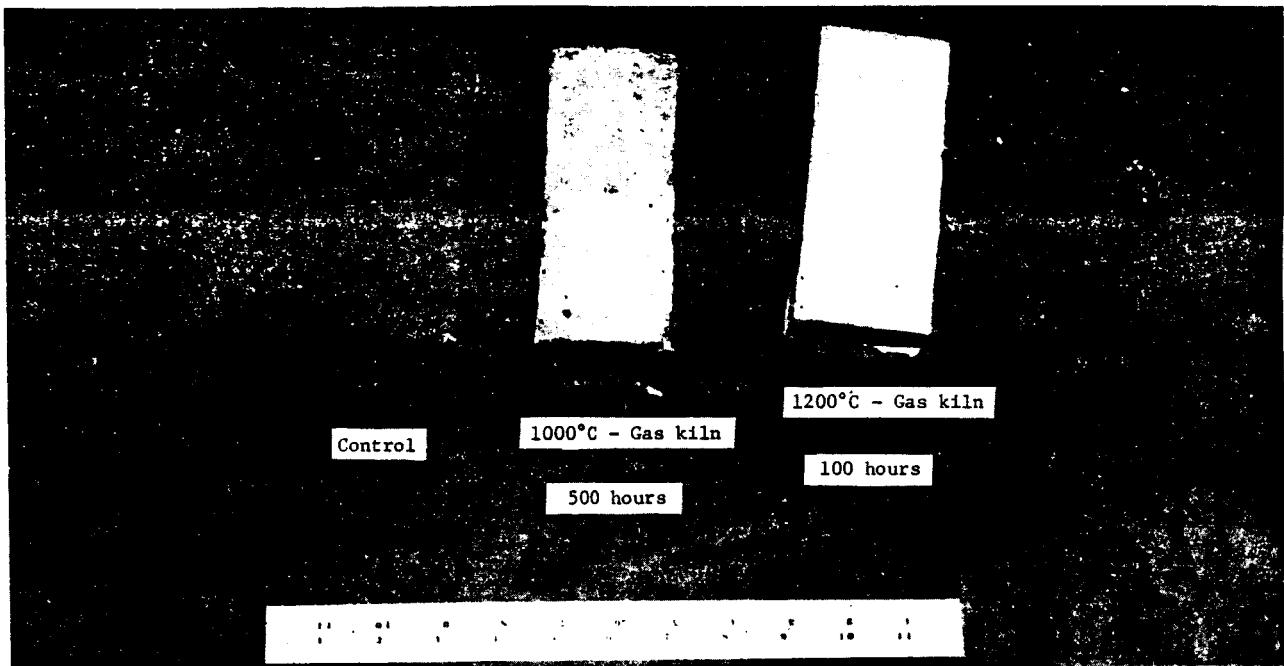


Figure 7. - Irish Refrasil fiber cake samples.

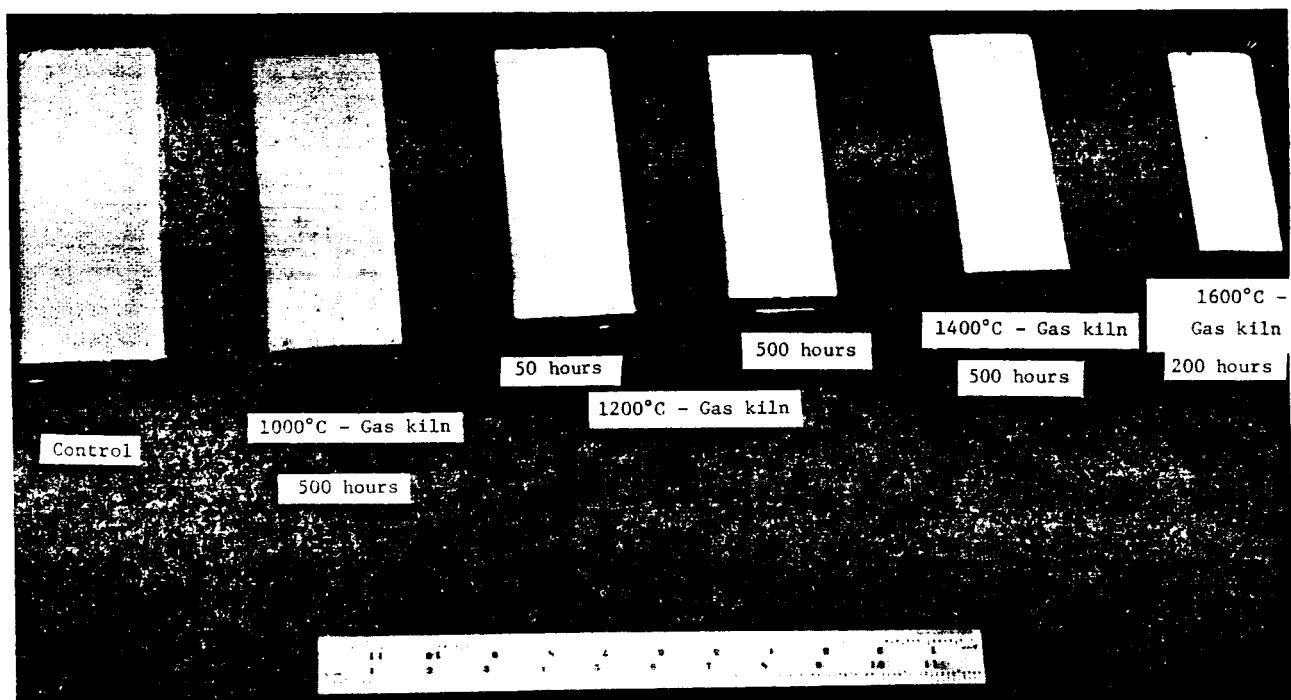


Figure 8. - Saffil Alumina HT fiber cake samples.

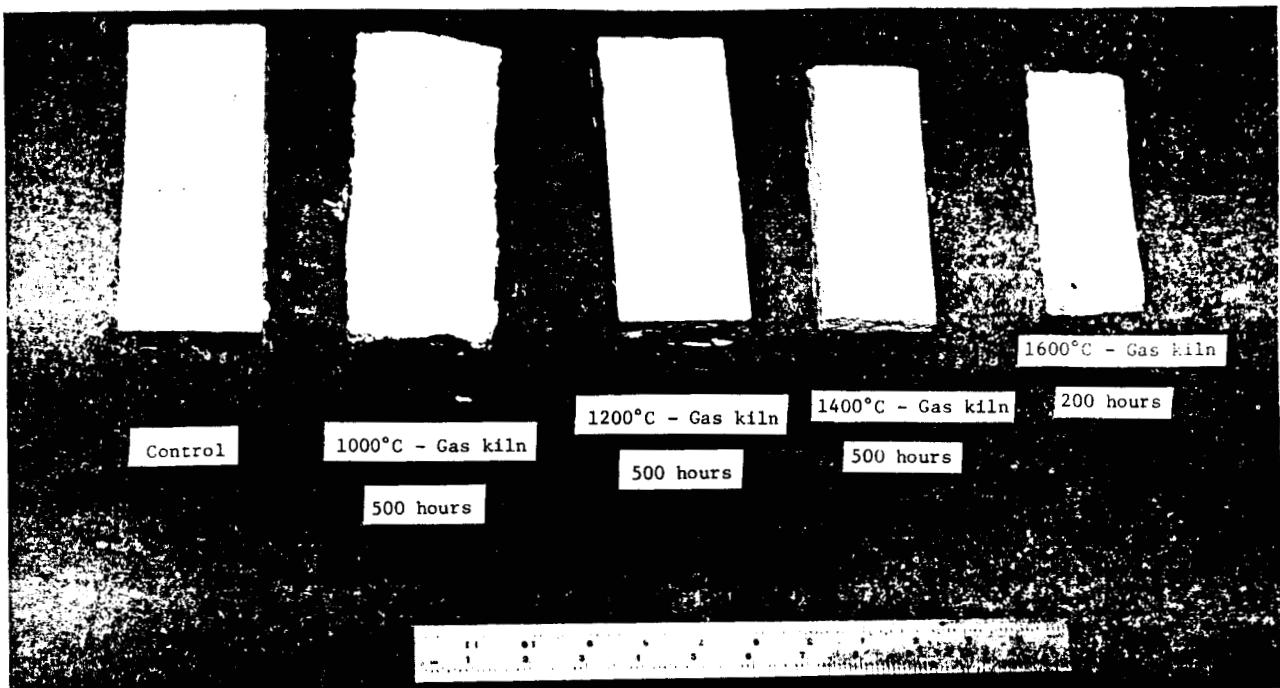


Figure 9. - Saffil Zirconia HT fiber cake samples.

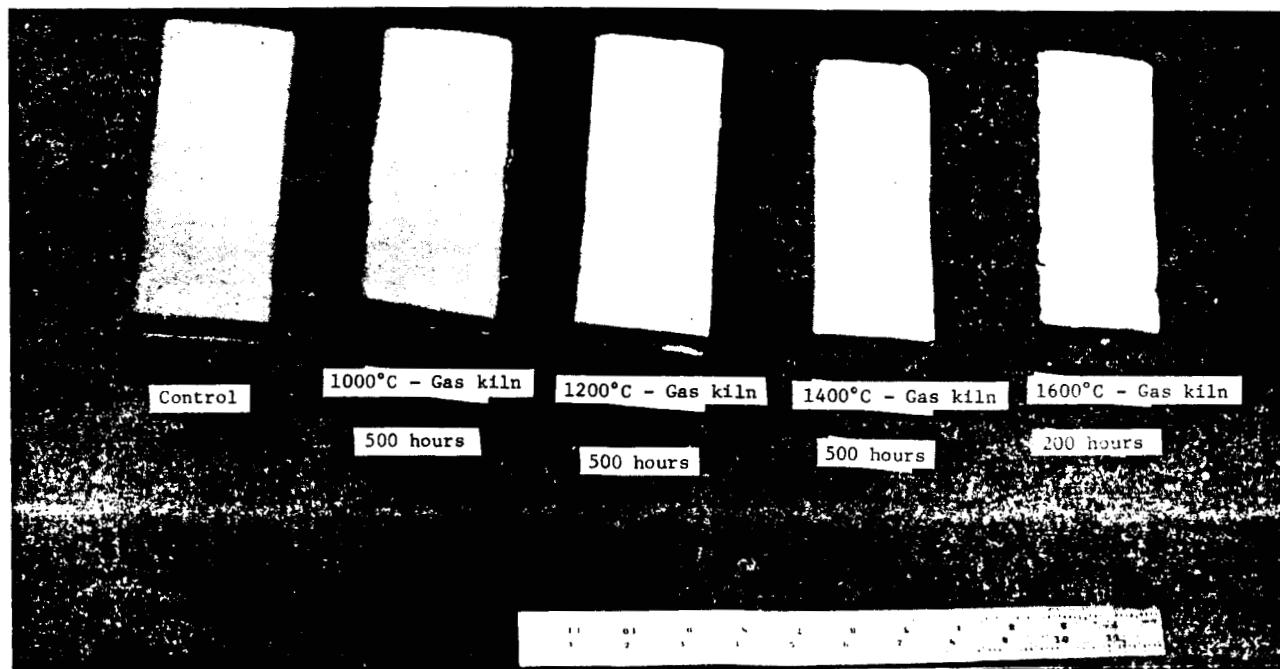


Figure 10. - B&W Mullite fiber cake samples.

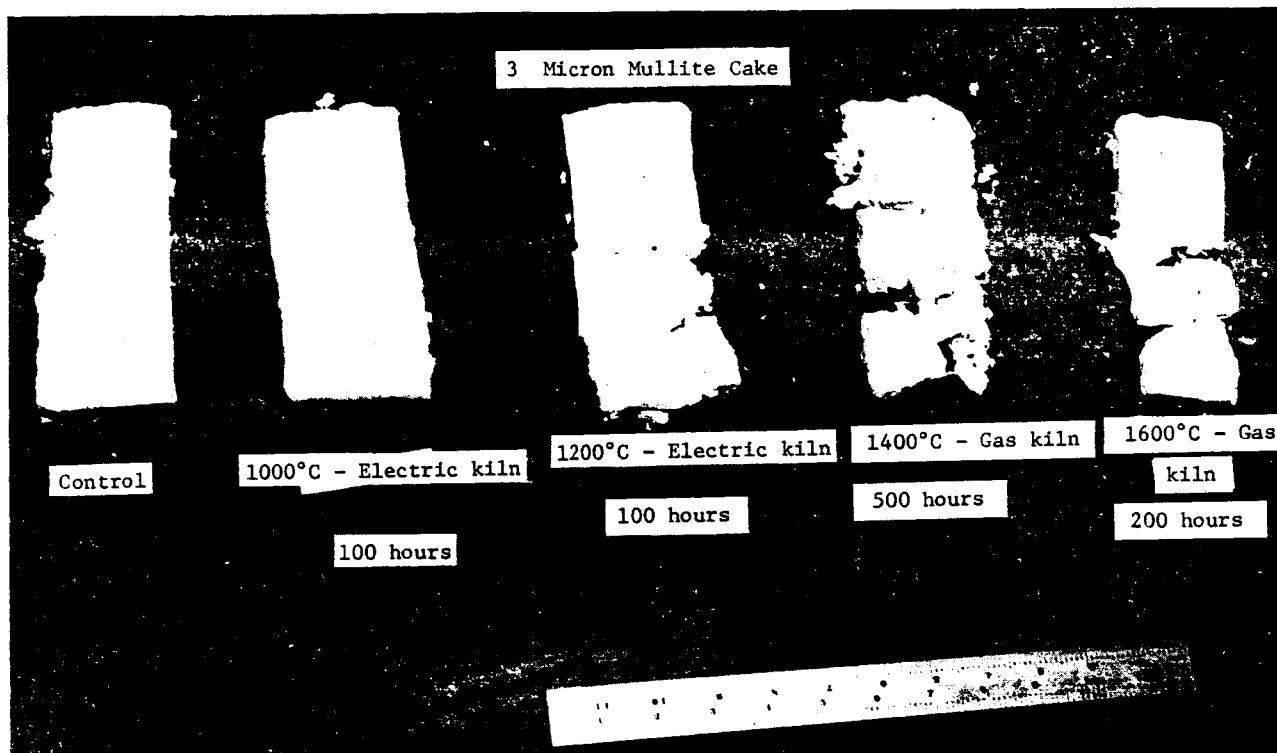


Figure 11.- 3 Micron Mullite fiber cake samples.

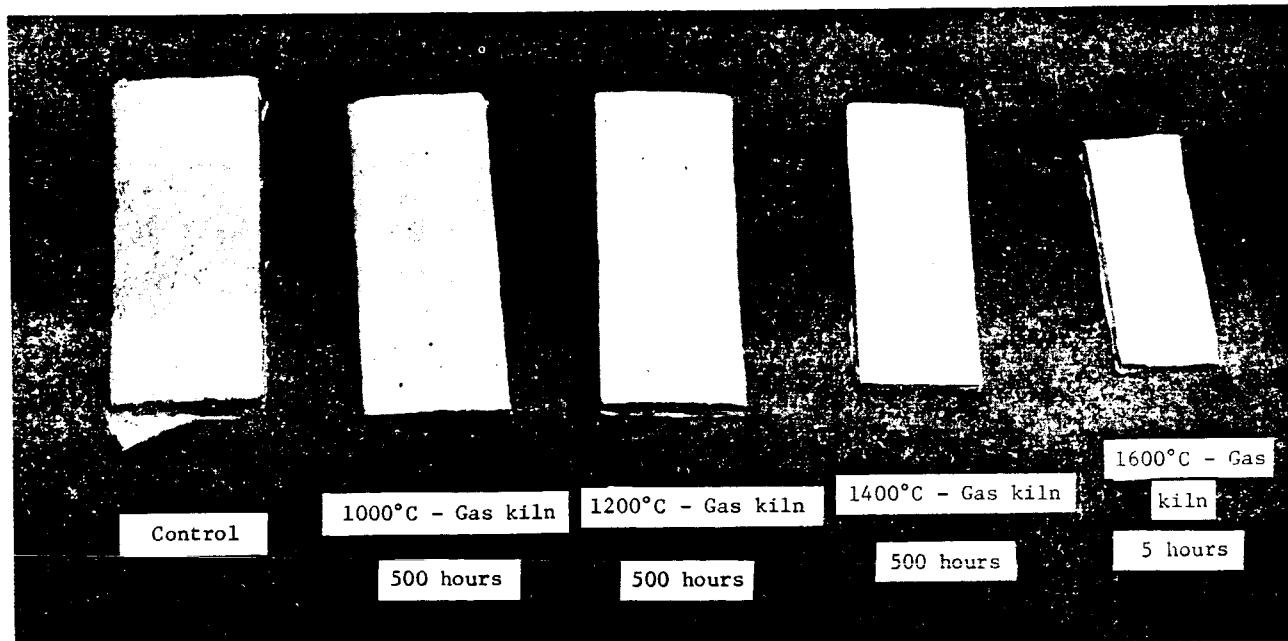


Figure 12.- Fiberfrax H fiber cake samples.

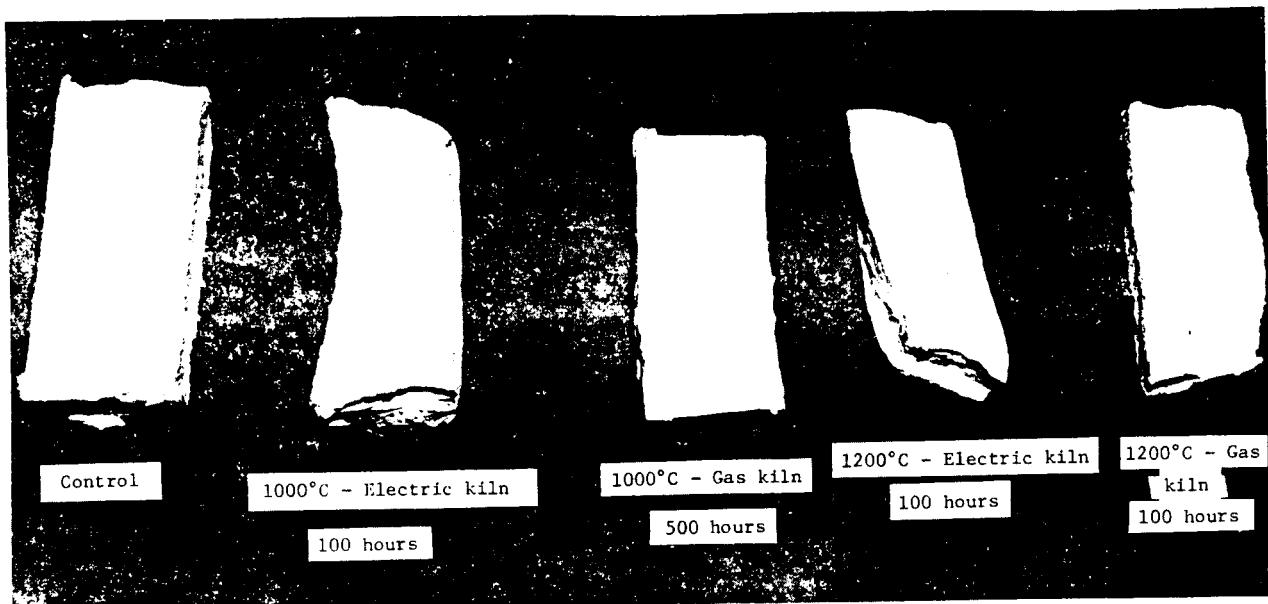


Figure 13.- Fiberfrax H blanket samples.

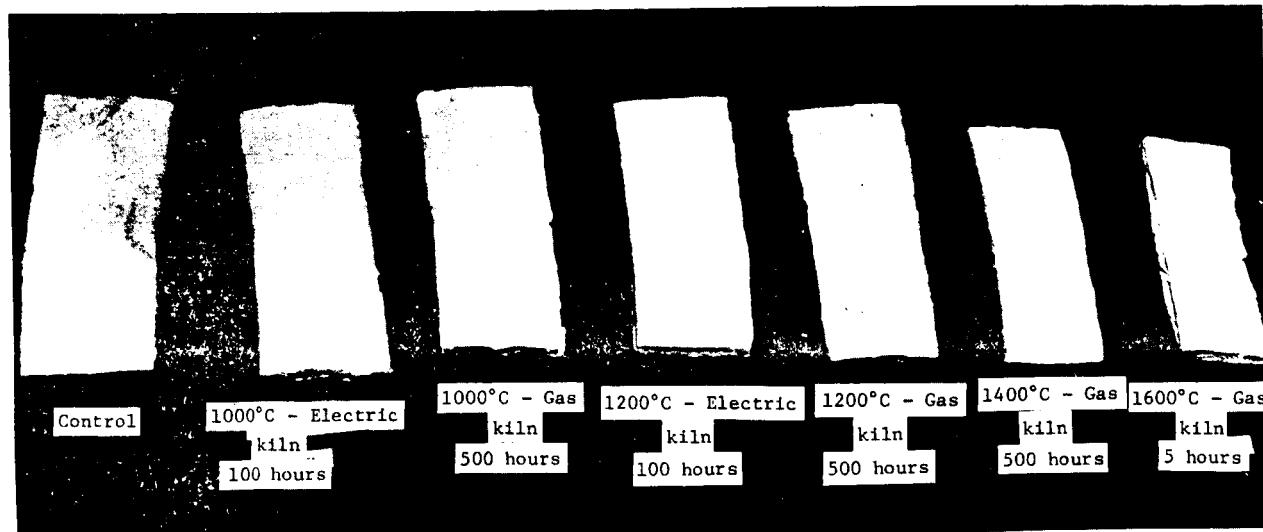


Figure 14.- Kaowool 1400 blanket samples.

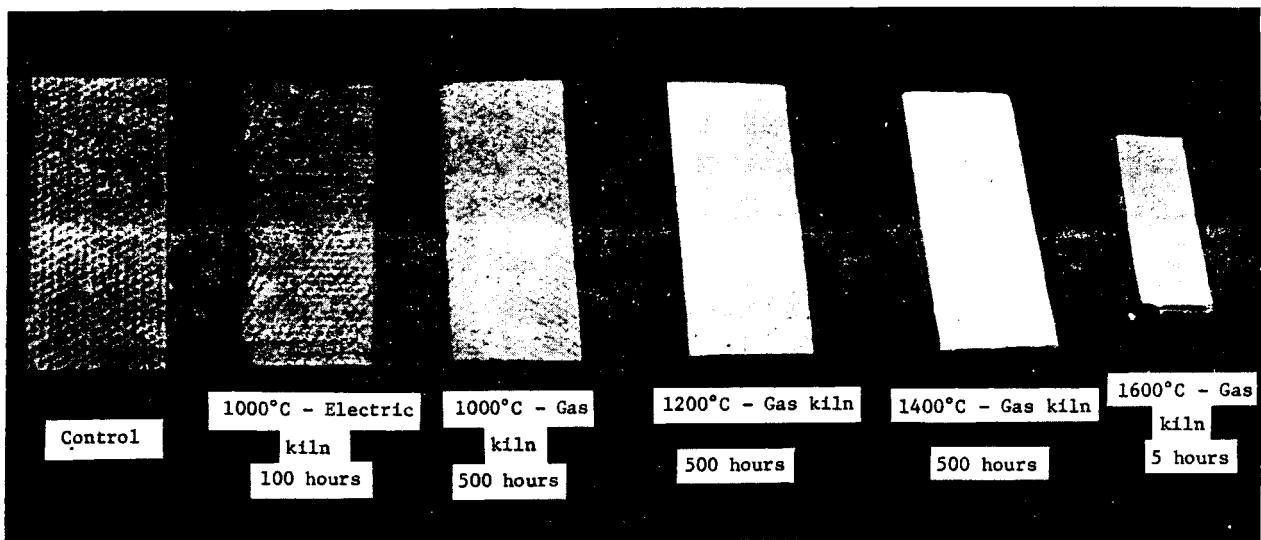


Figure 15.- Fiberchrome blanket samples.

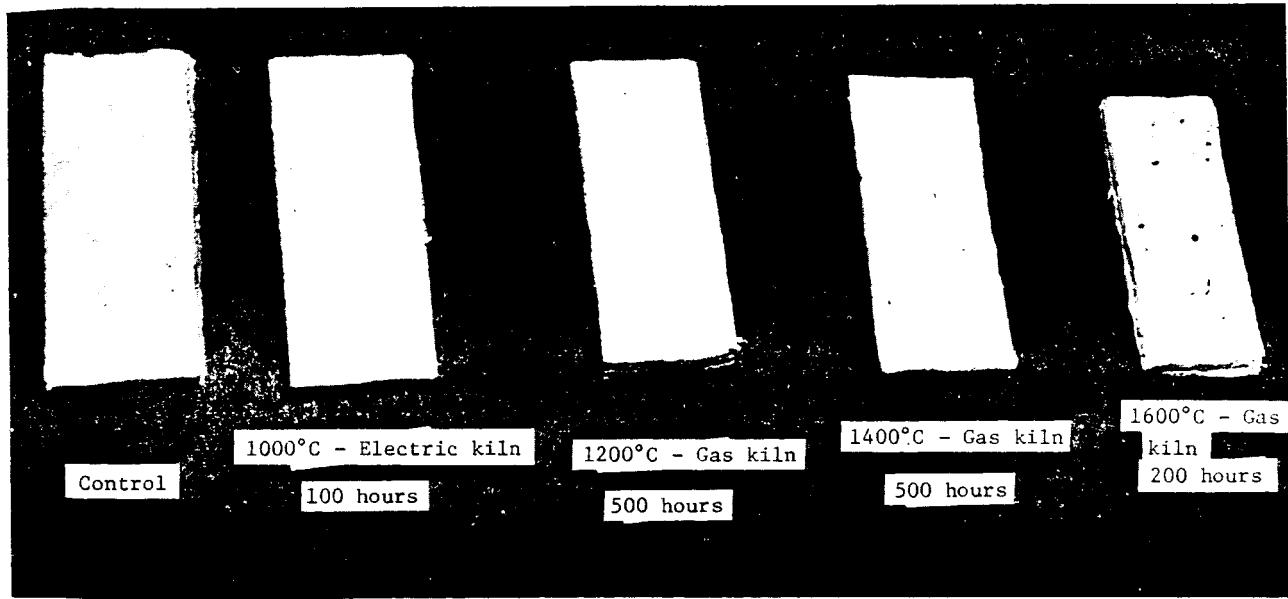


Figure 16.- Saffil Alumina HT blanket samples.

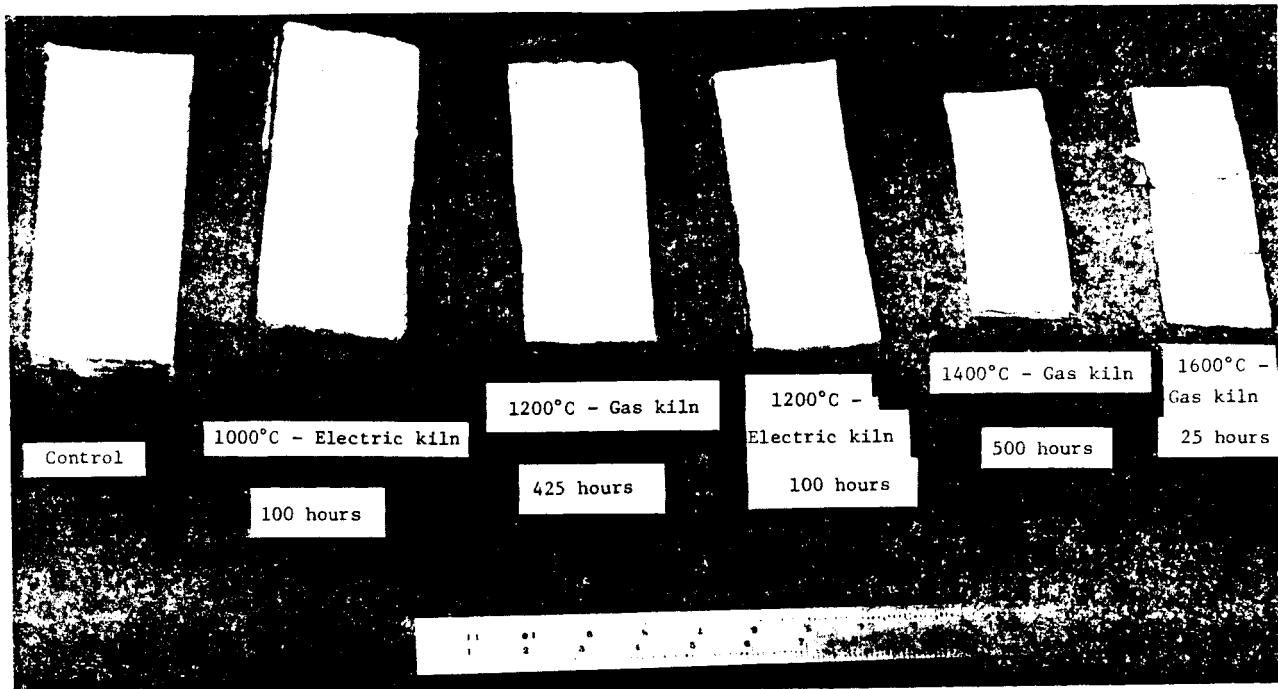


Figure 17.- Saffil Zirconia HT blanket samples.

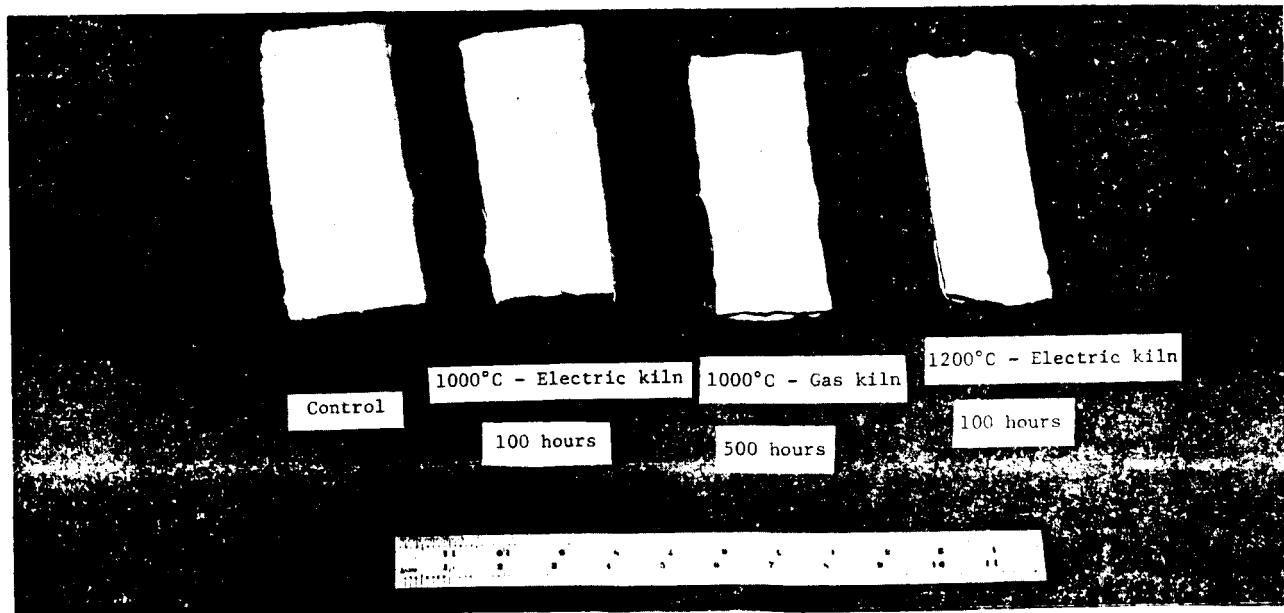


Figure 18.- Microquartz blanket samples.

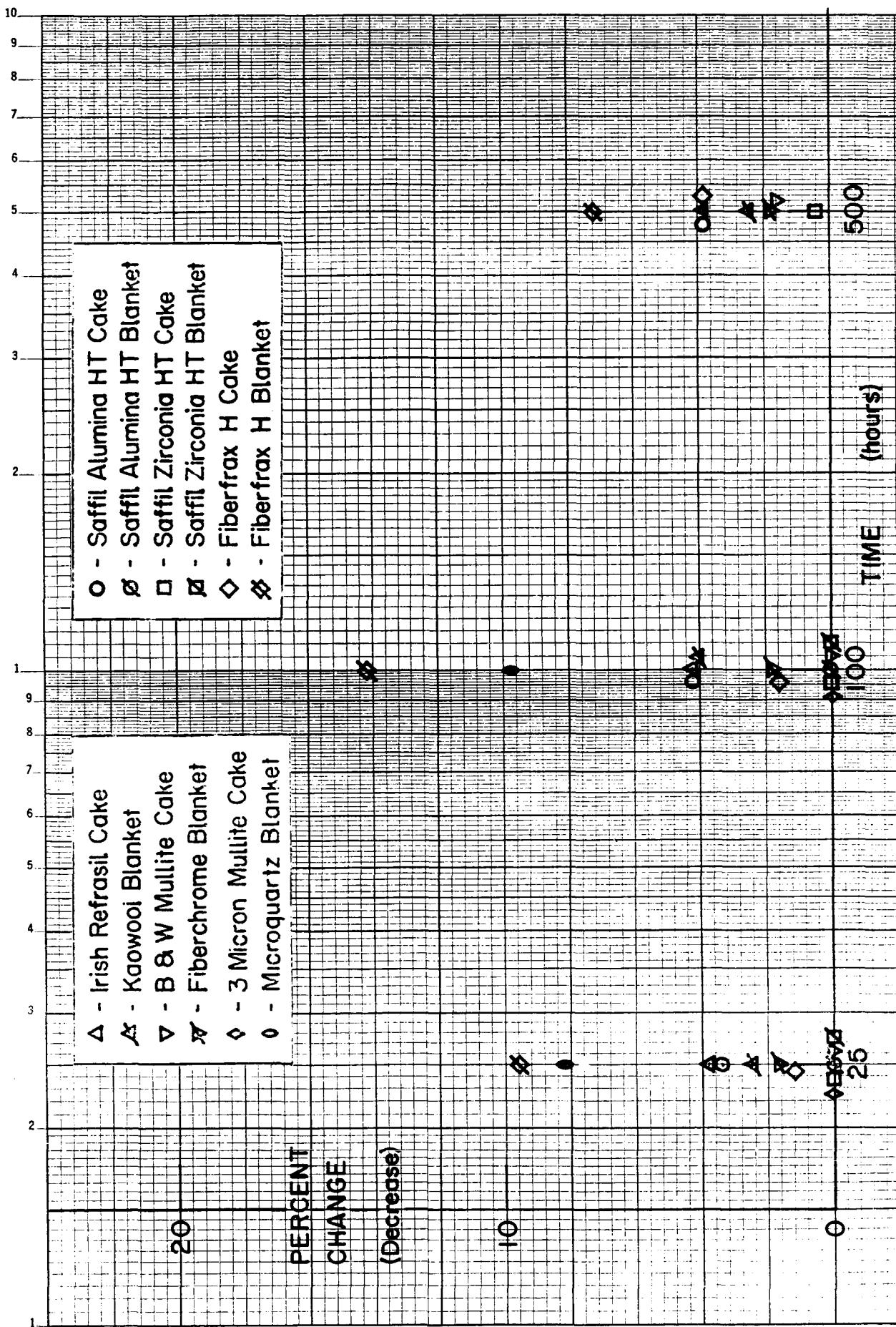


Figure 19.—Change in length of fiber cakes and blankets during 1000°C (1832°F) exposure.

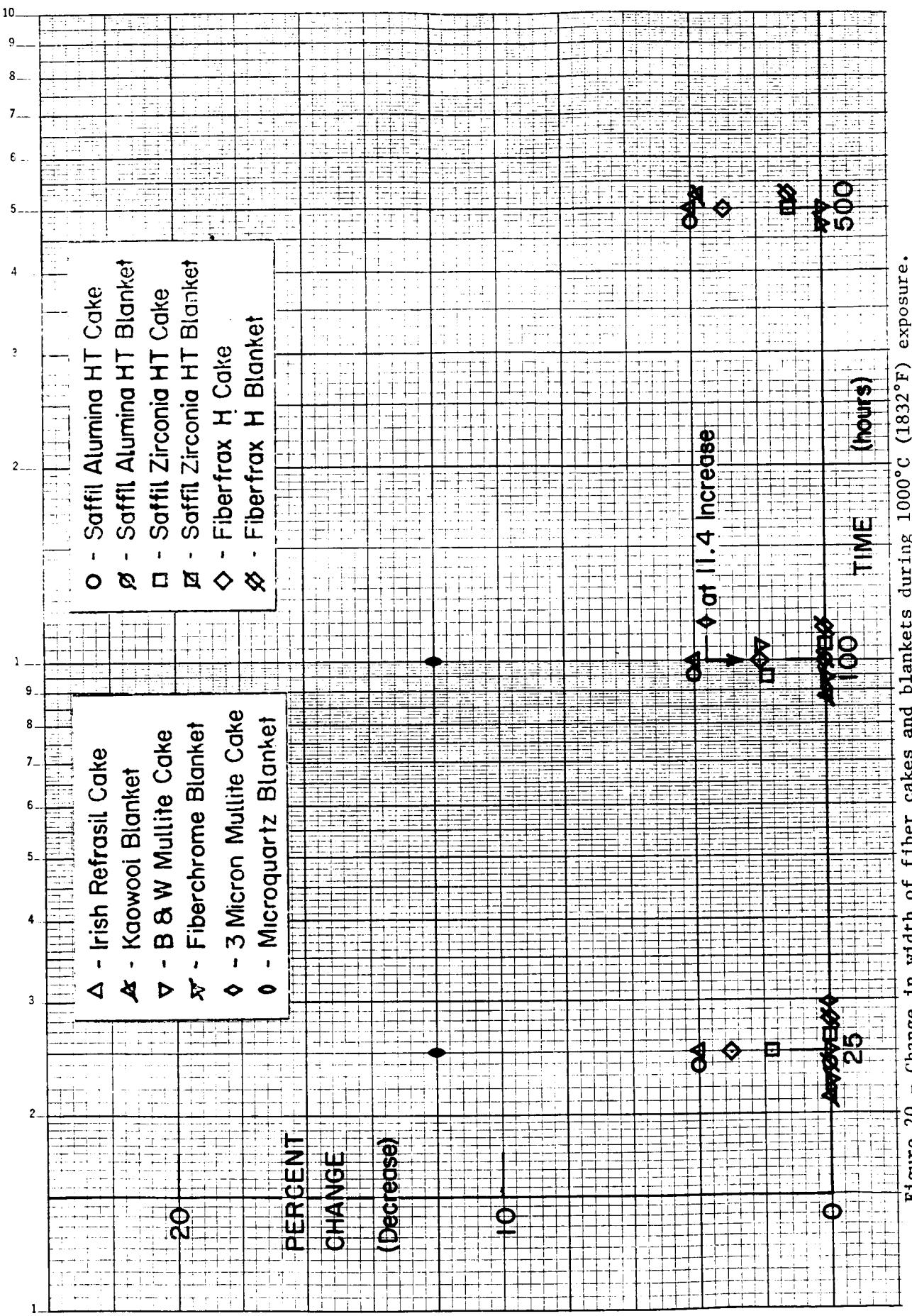


Figure 20. - Change in width of fiber cakes and blankets during 1000°C (1832°F) exposure.

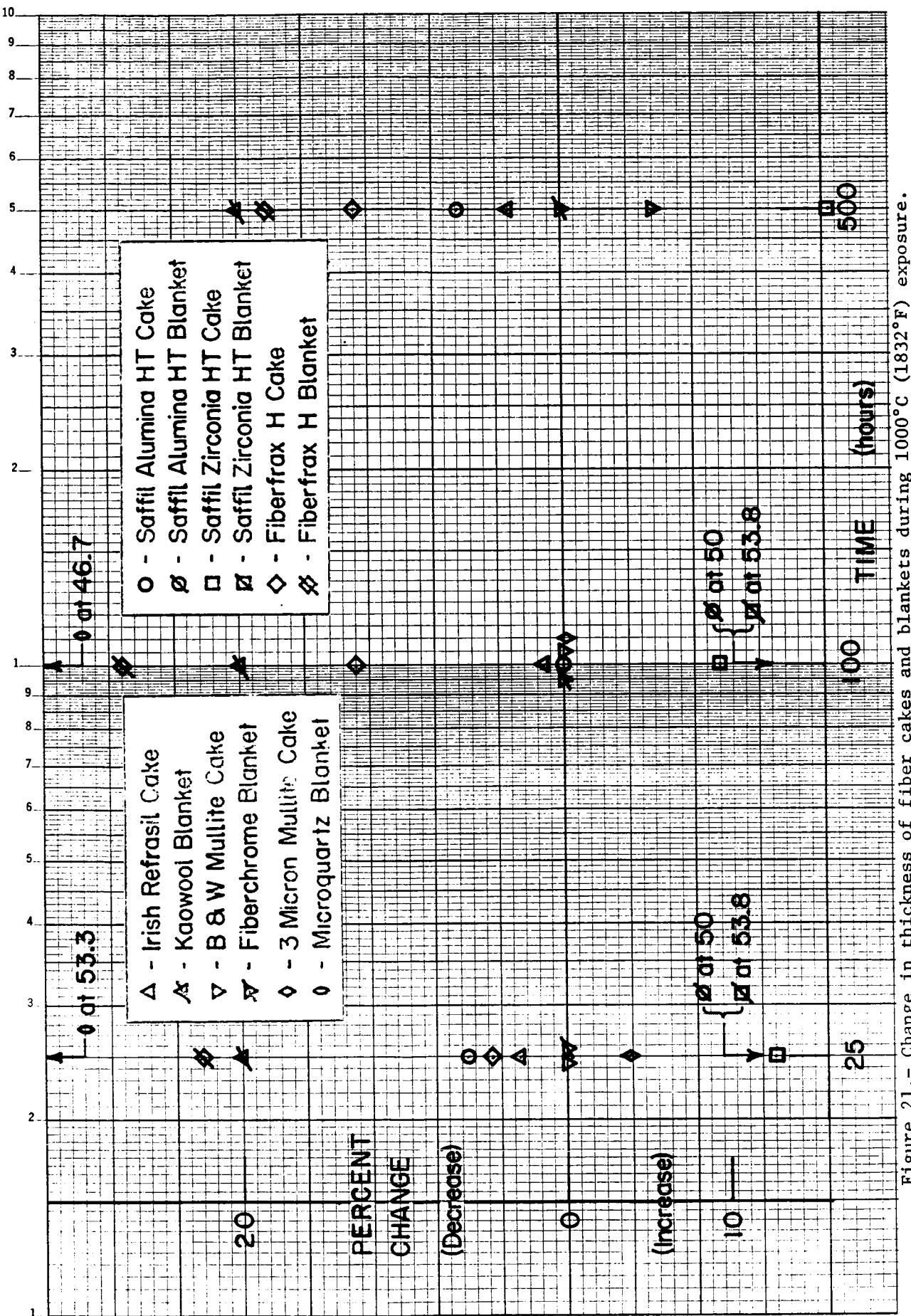


Figure 21.- Change in thickness of fiber cakes and blankets during 1000°C (1832°F) exposure.

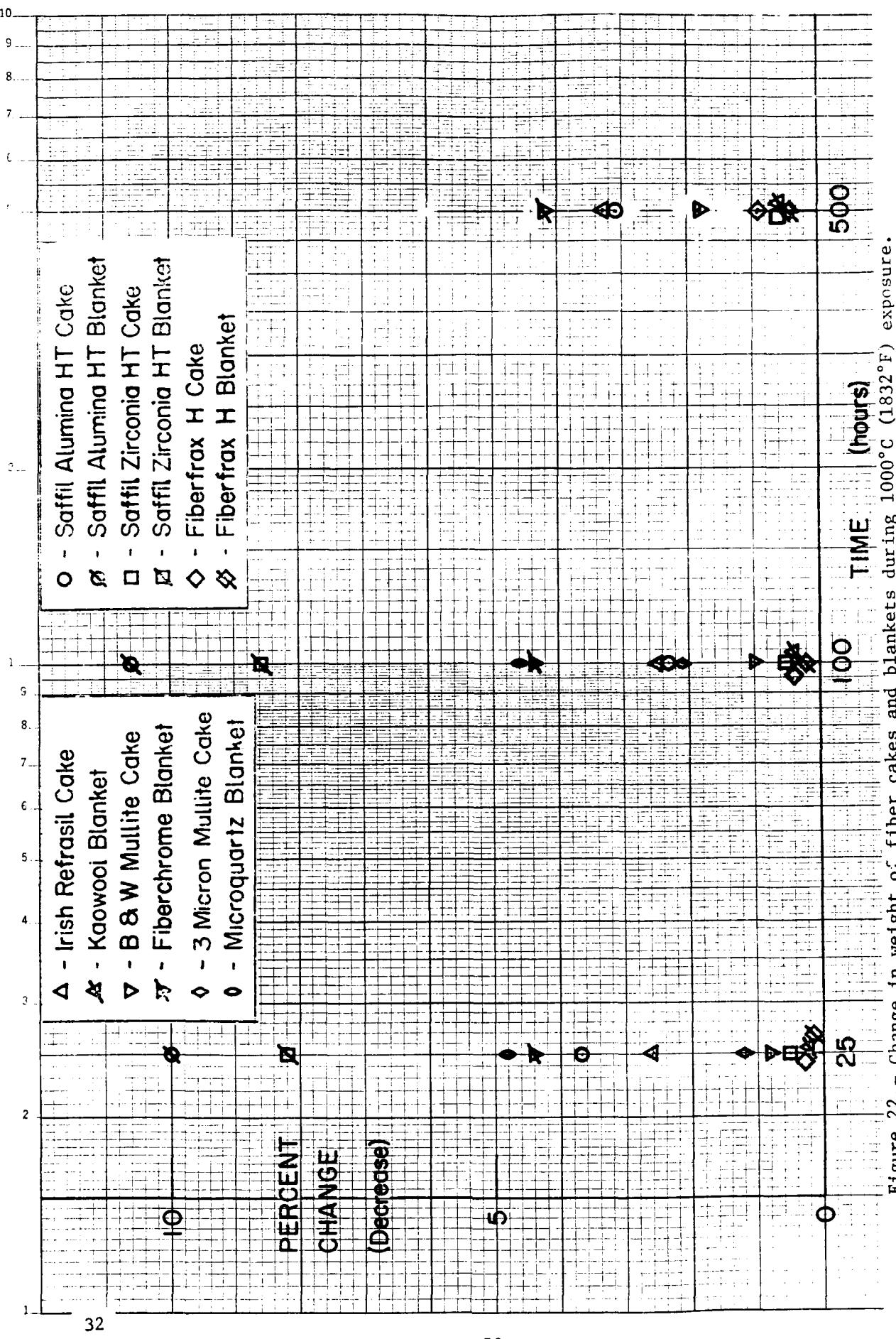


Figure 22.—Change in weight of fiber cakes and blankets during 1000°C (1832°F) exposure.

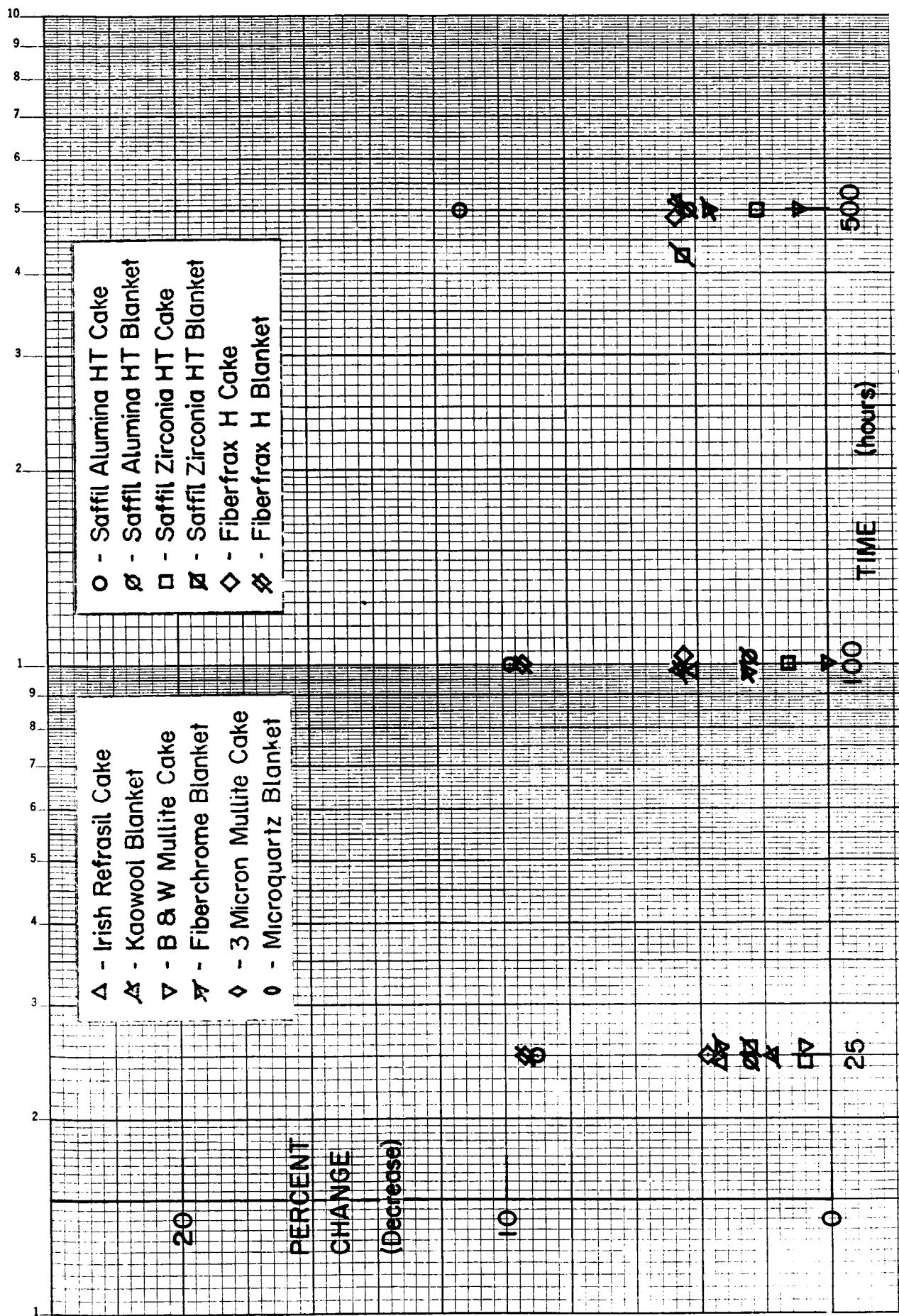


Figure 23.0 - Change in length of fiber cakes and blankets during 1200°C (2192°F) exposure.

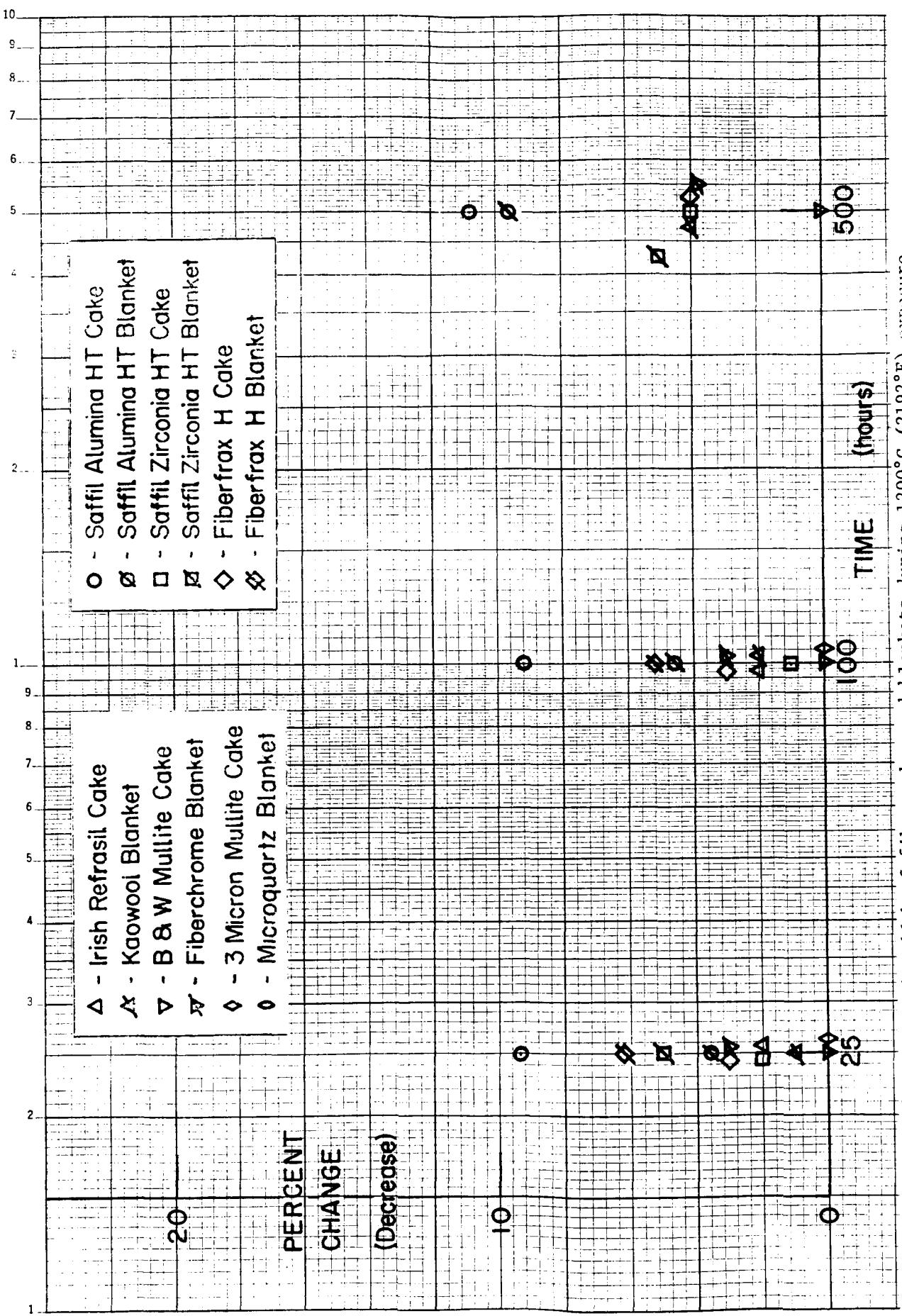


Figure 24.- Change in width of fiber cakes and blankets during 1200°C (2192°F) exposure.

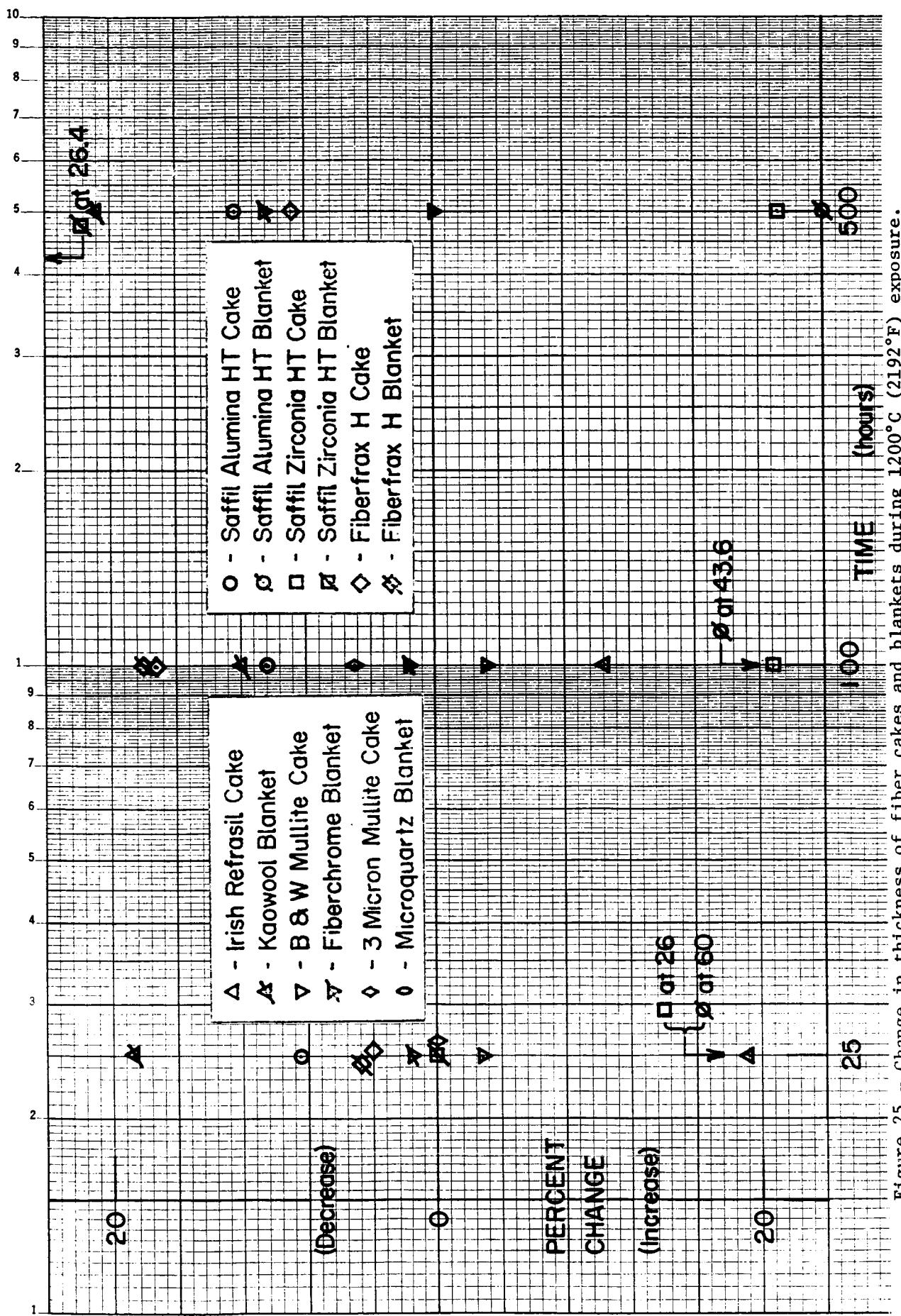


Figure 25. - Change in thickness of fiber cakes and blankets during 1200°C (2192°F) exposure.

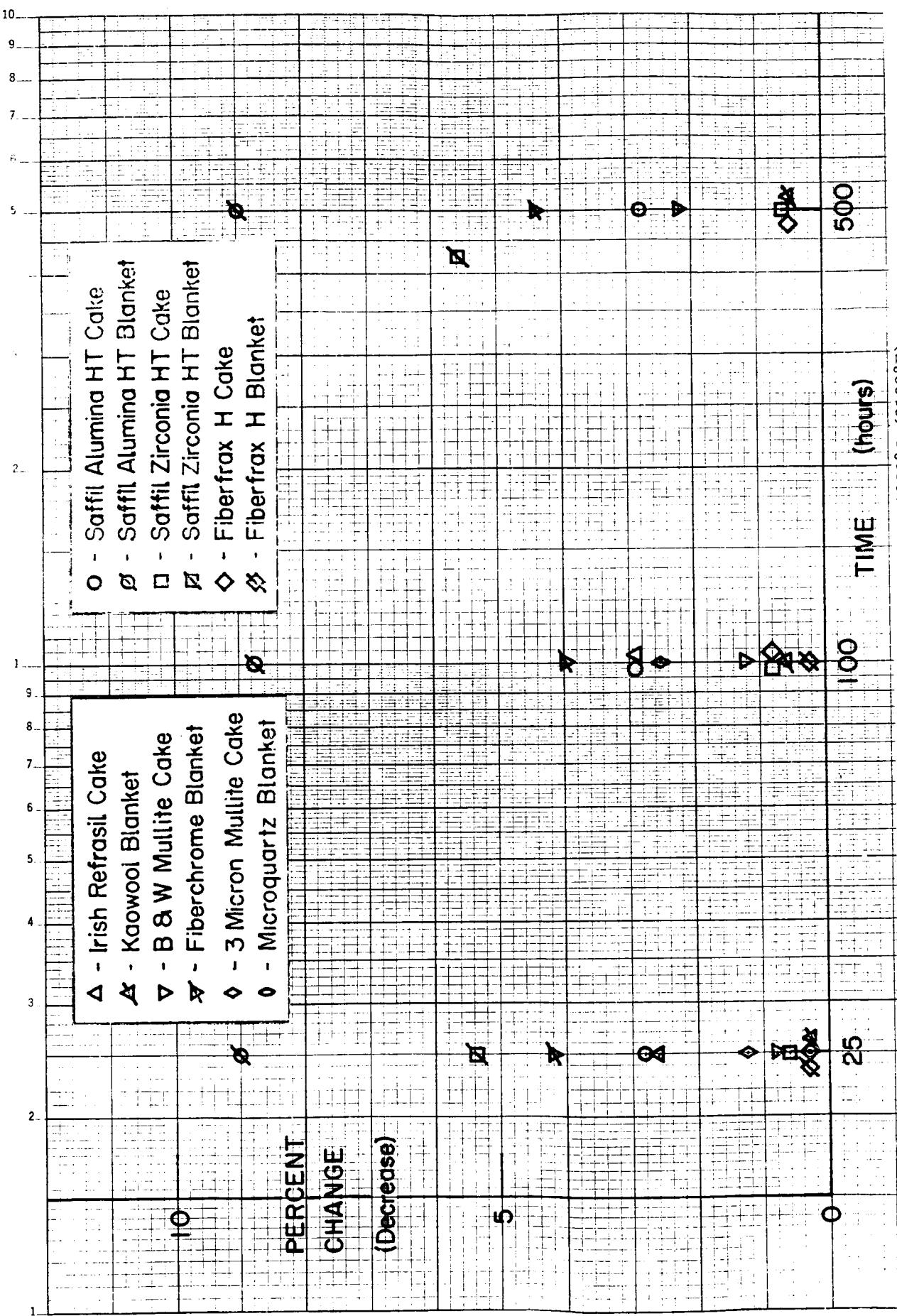


Figure 26.—Change in weight of fiber cakes and blankets during 1200°C (2192°F) exposure.

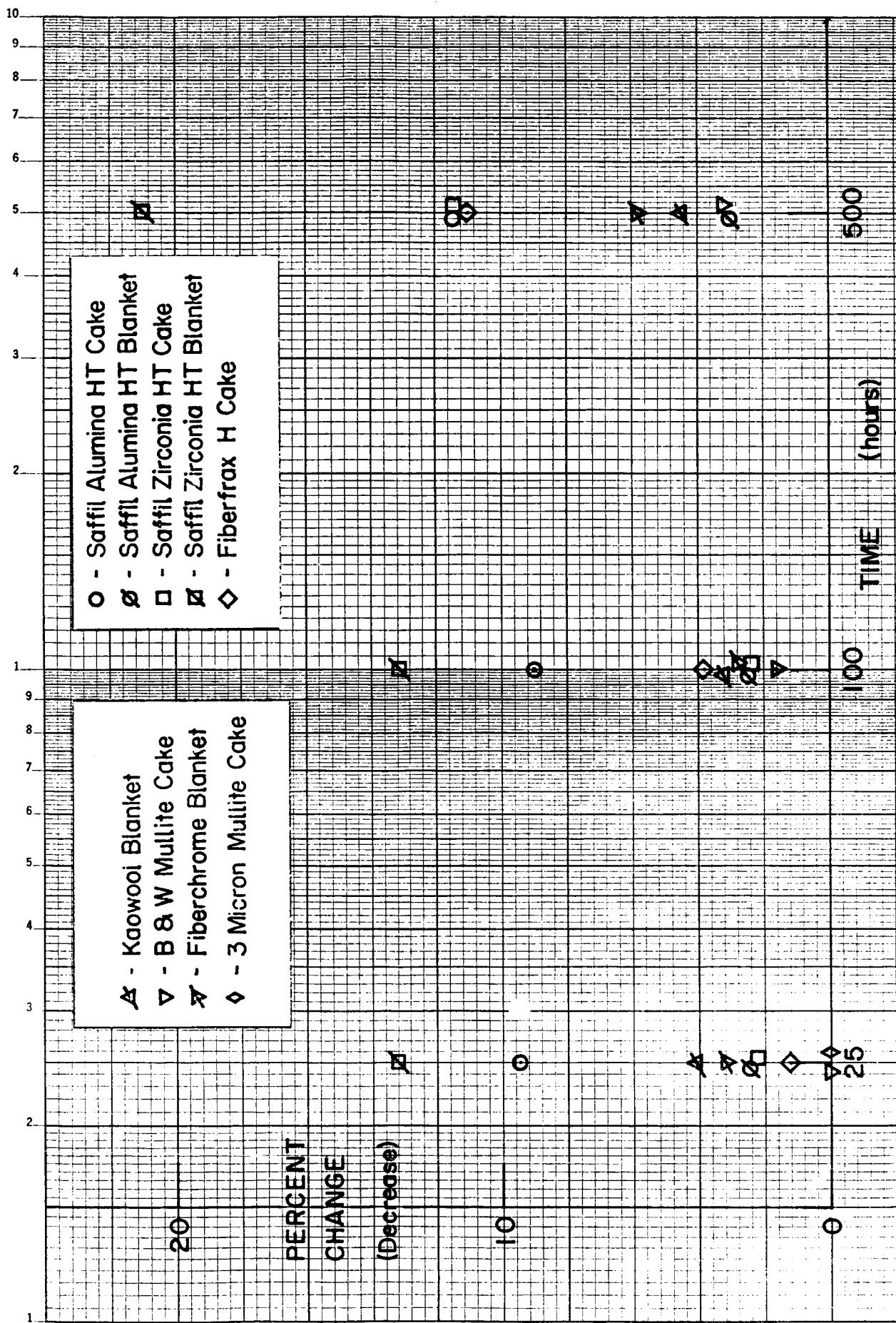


Figure 27.- Change in length of fiber cakes and blankets during 1400°C (2552°F) exposure.

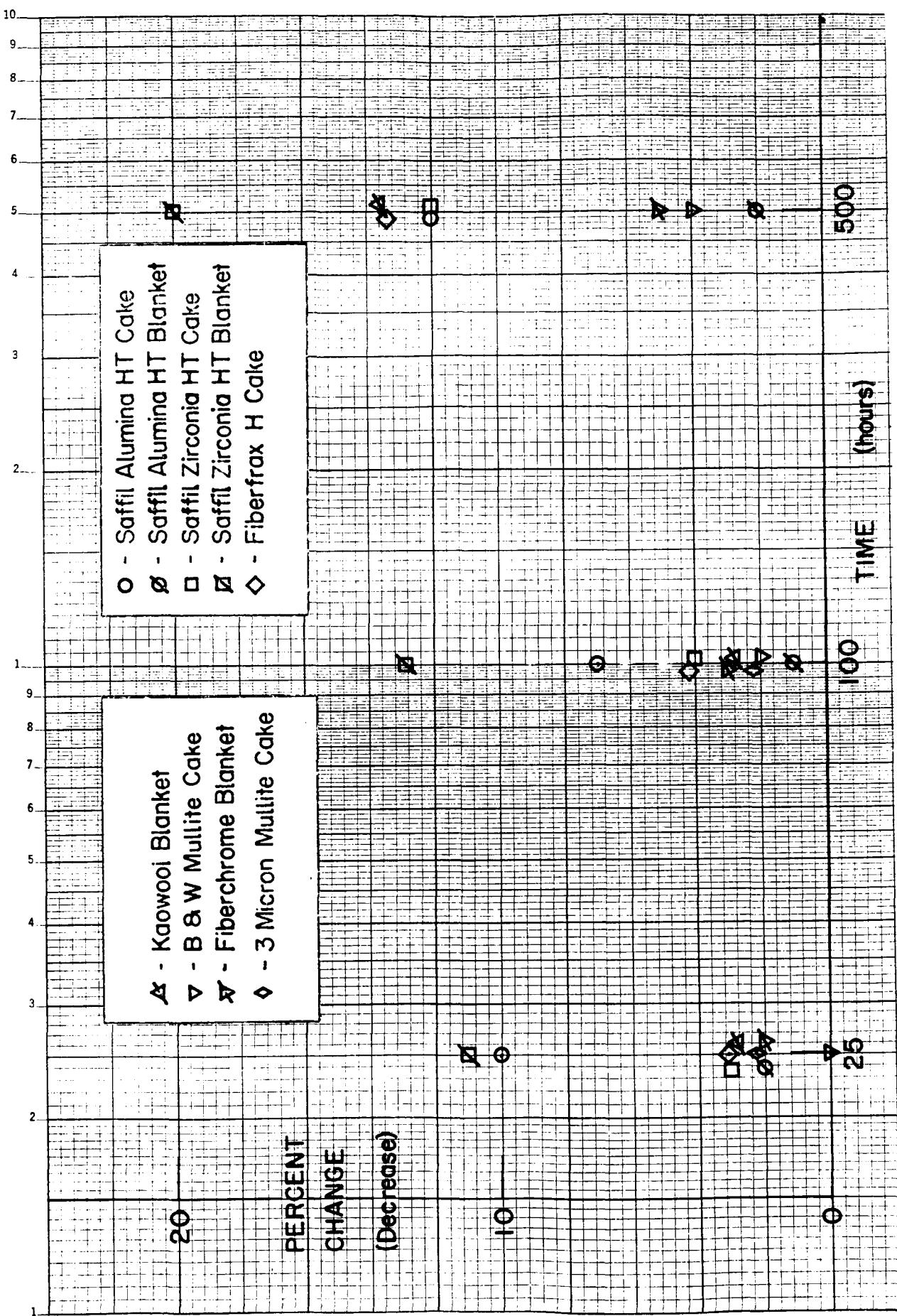


Figure 28.— Change in width of fiber cakes and blankets during 1400°C (2552°F) exposure.

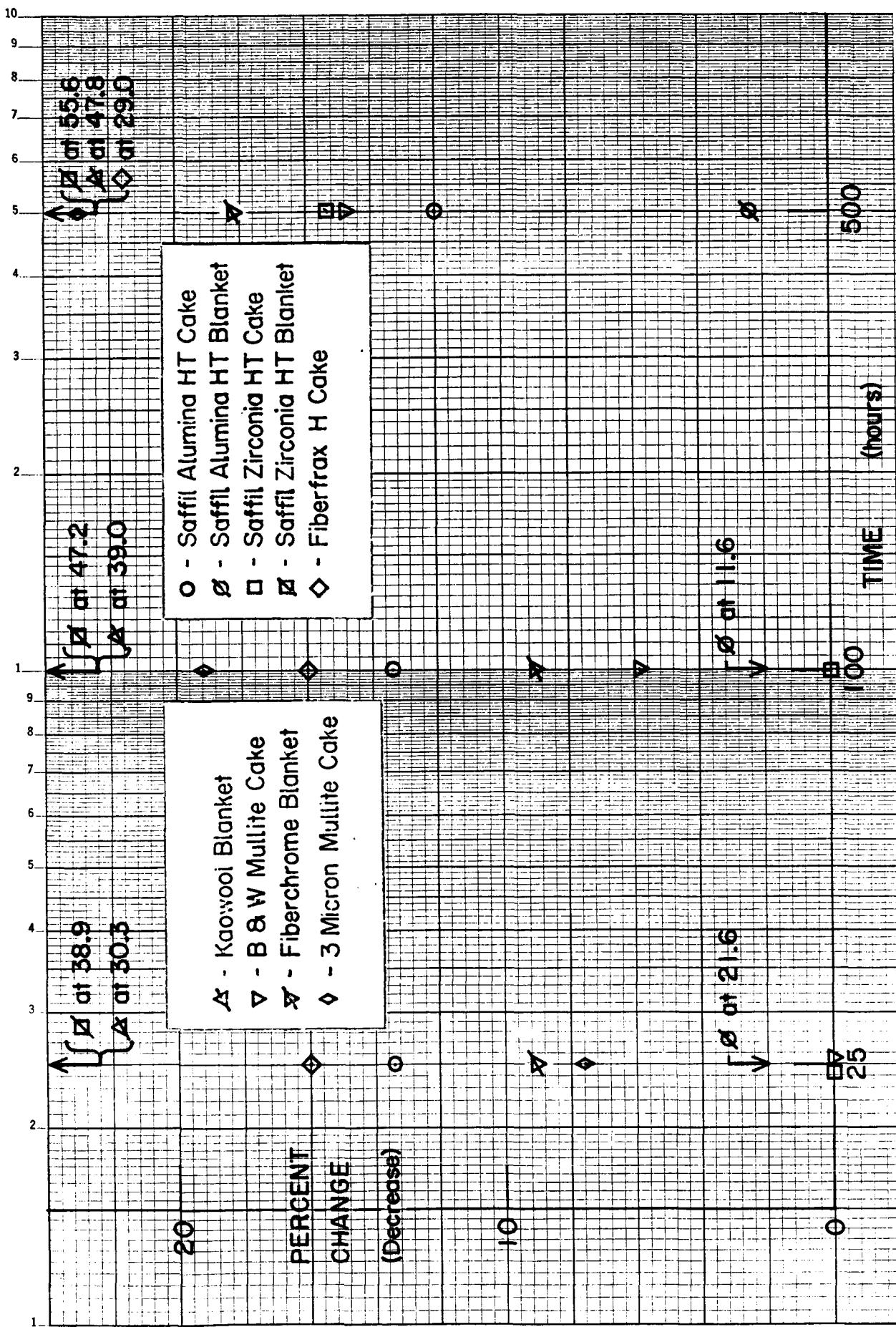


Figure 29.— Change in thickness of fiber cakes and blankets during 1400°C (2552°F) exposure.

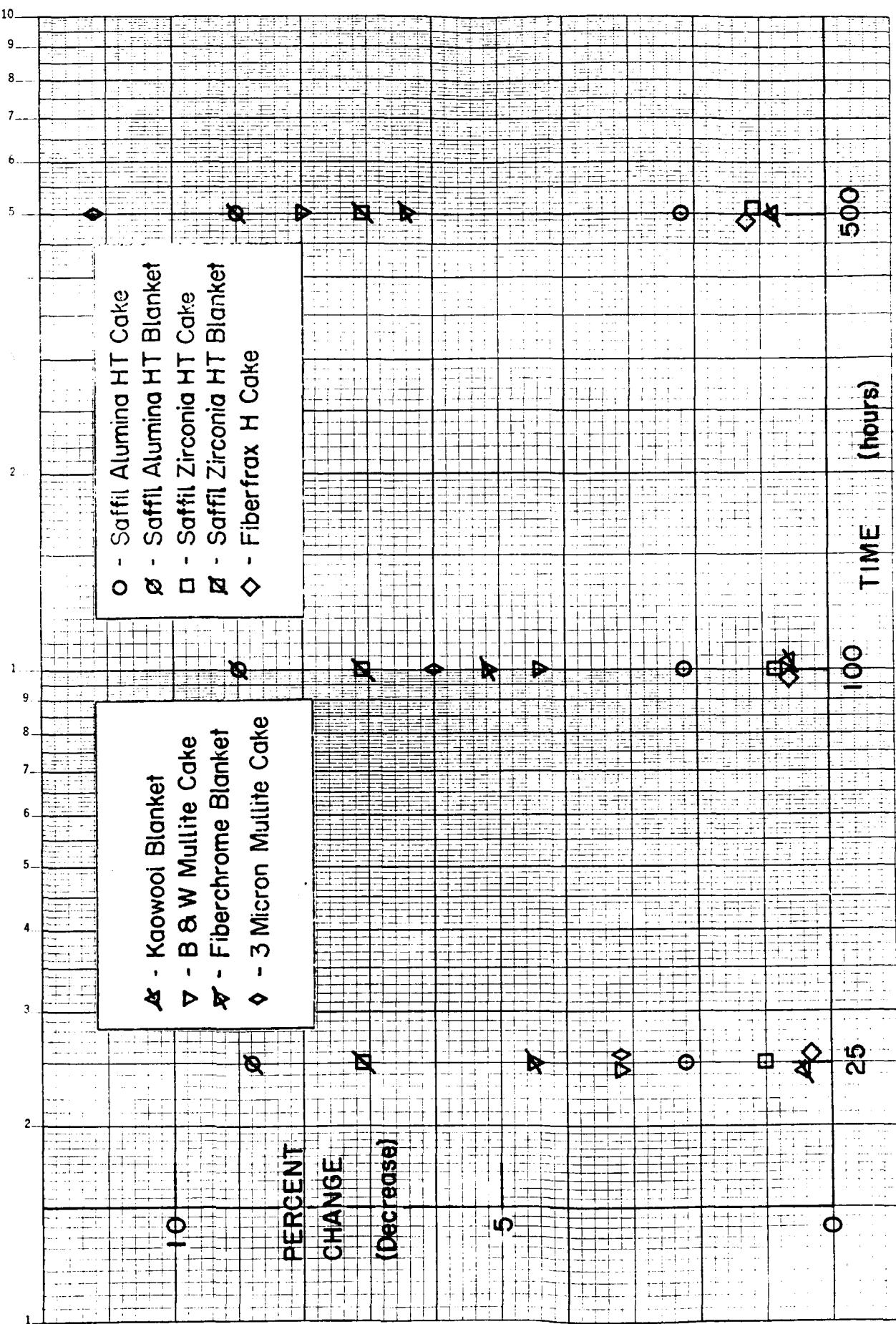


Figure 30.- Change in weight of fiber cakes and blankets during 1400°C (2552°F) exposure.

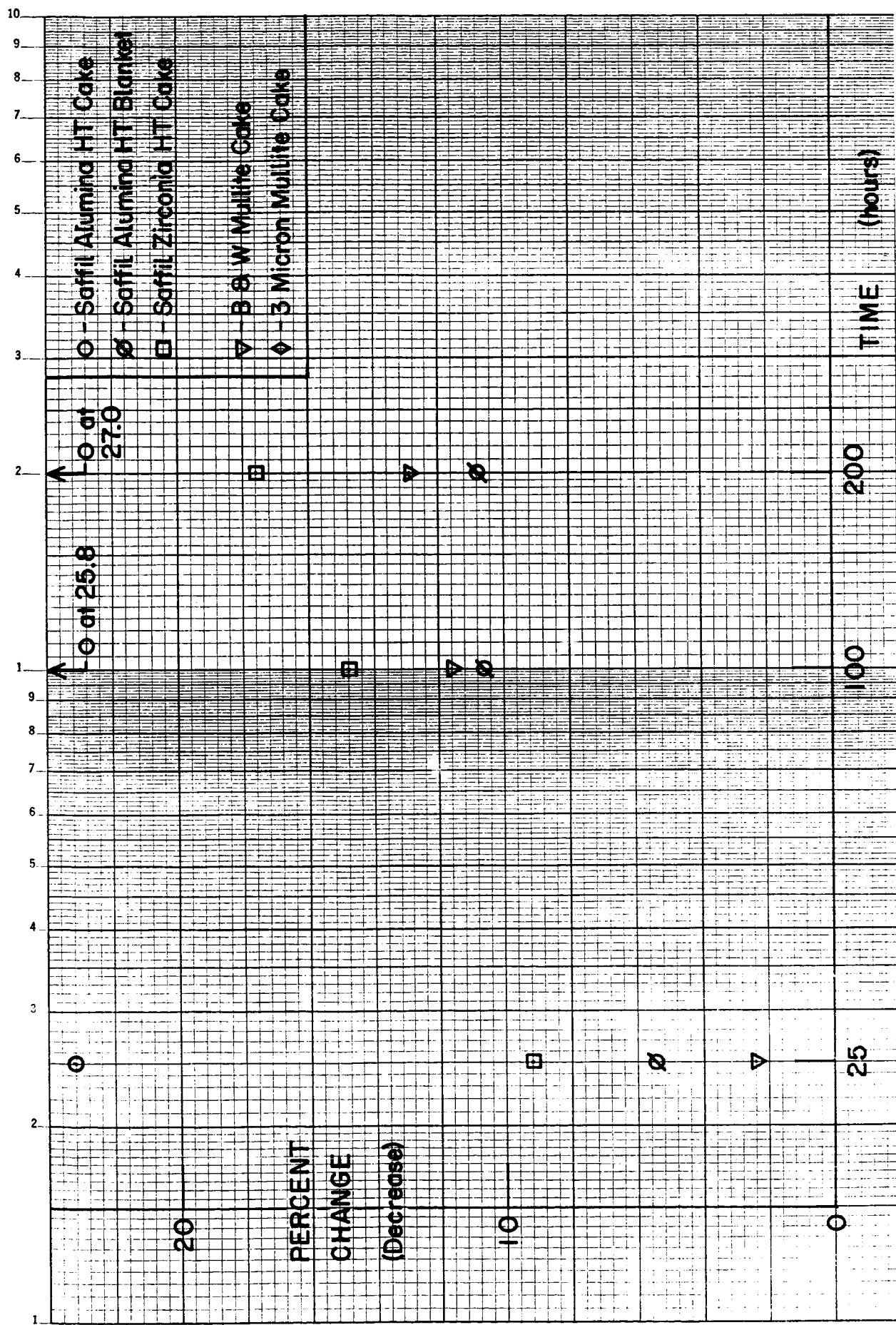


Figure 31.— Change in length of fiber cakes and blankets during 1600°C (2912°F) exposure.

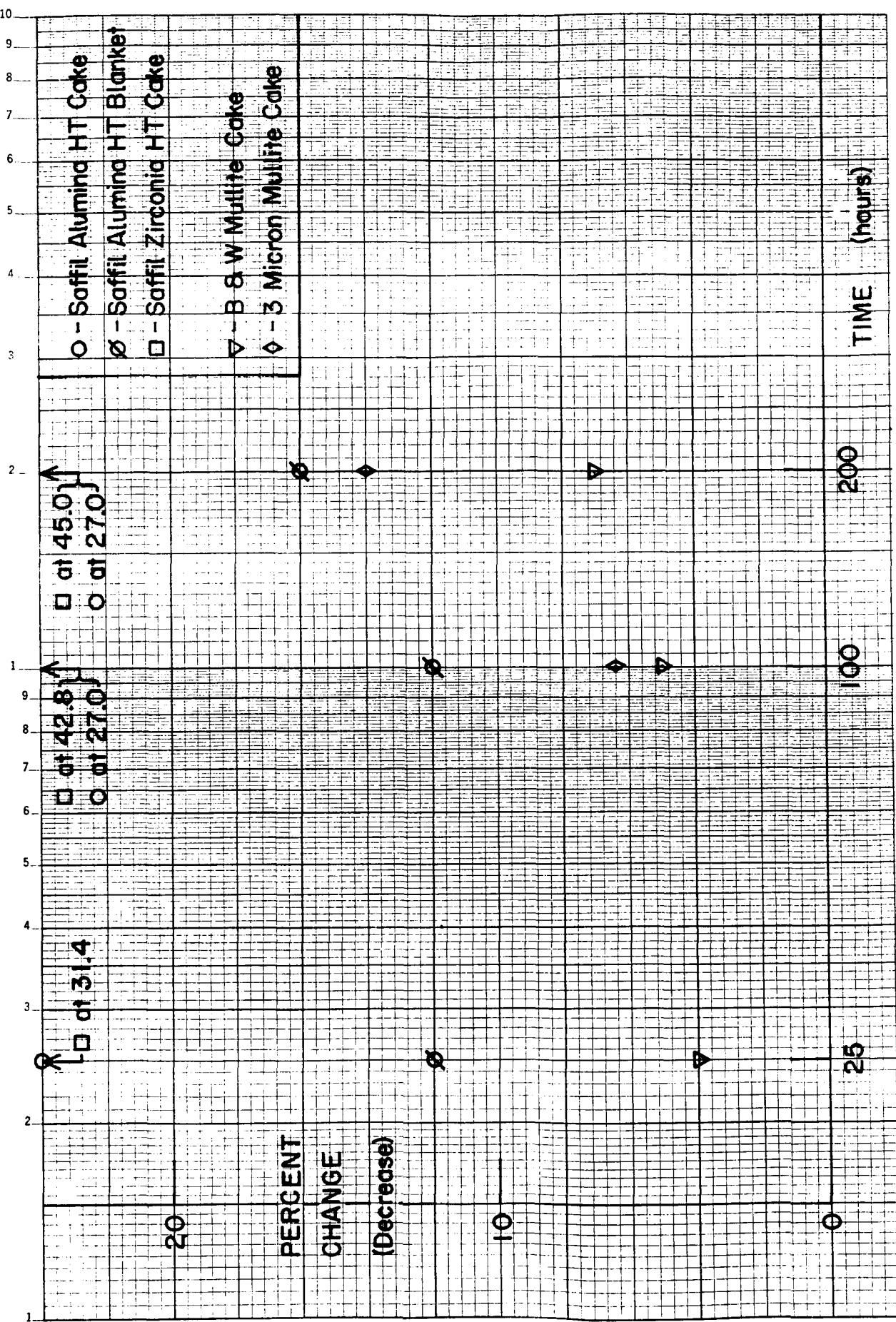


Figure 32. - Change in width of fiber cakes and blankets during 1600°C (2912°F) exposure.

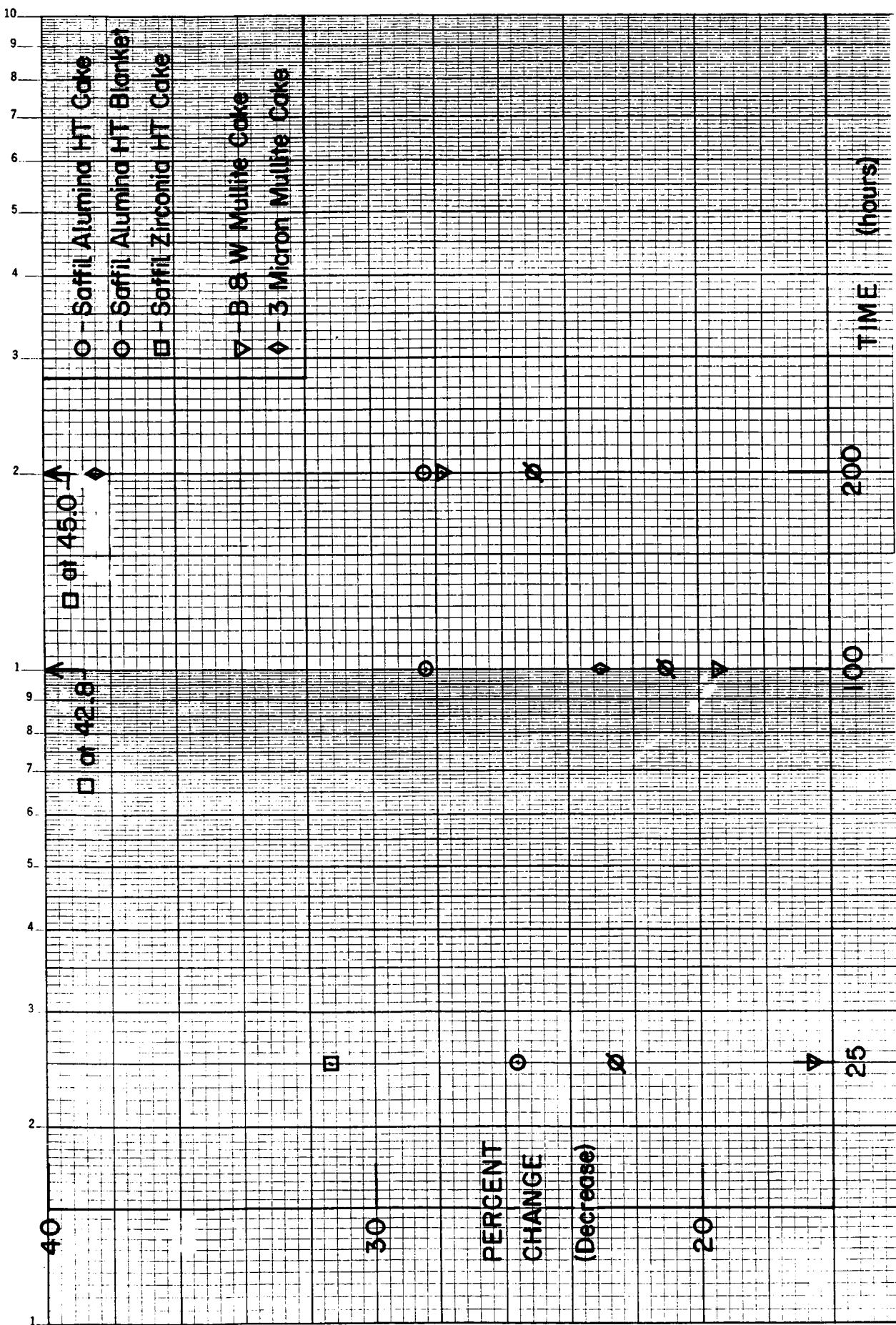


Figure 33.- Change in thickness of fiber cakes and blankets during 1600°C (2912°F) exposure.

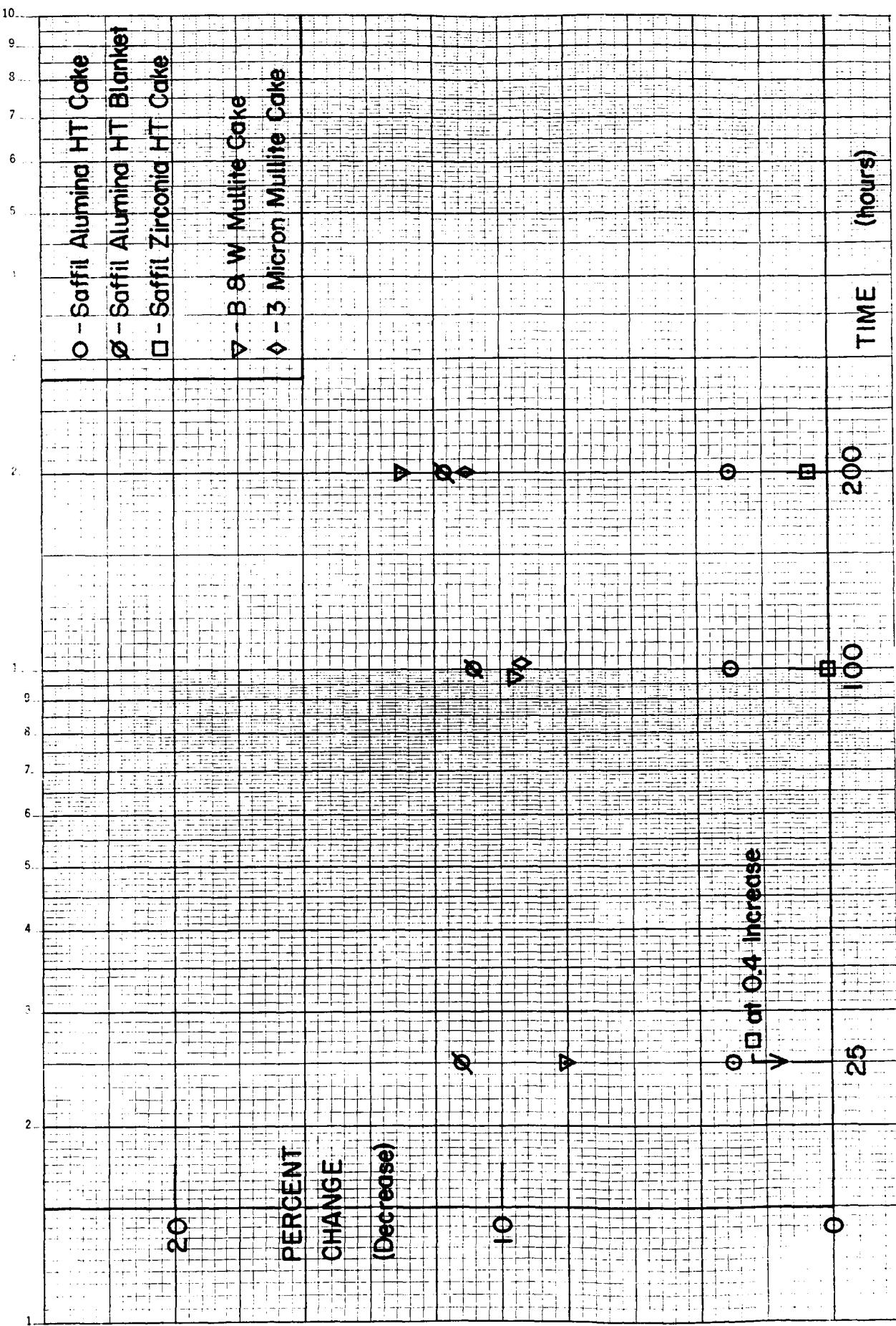


Figure 34. - Change in weight of fiber cakes and blankets during 1600°C (2912°F) exposure.

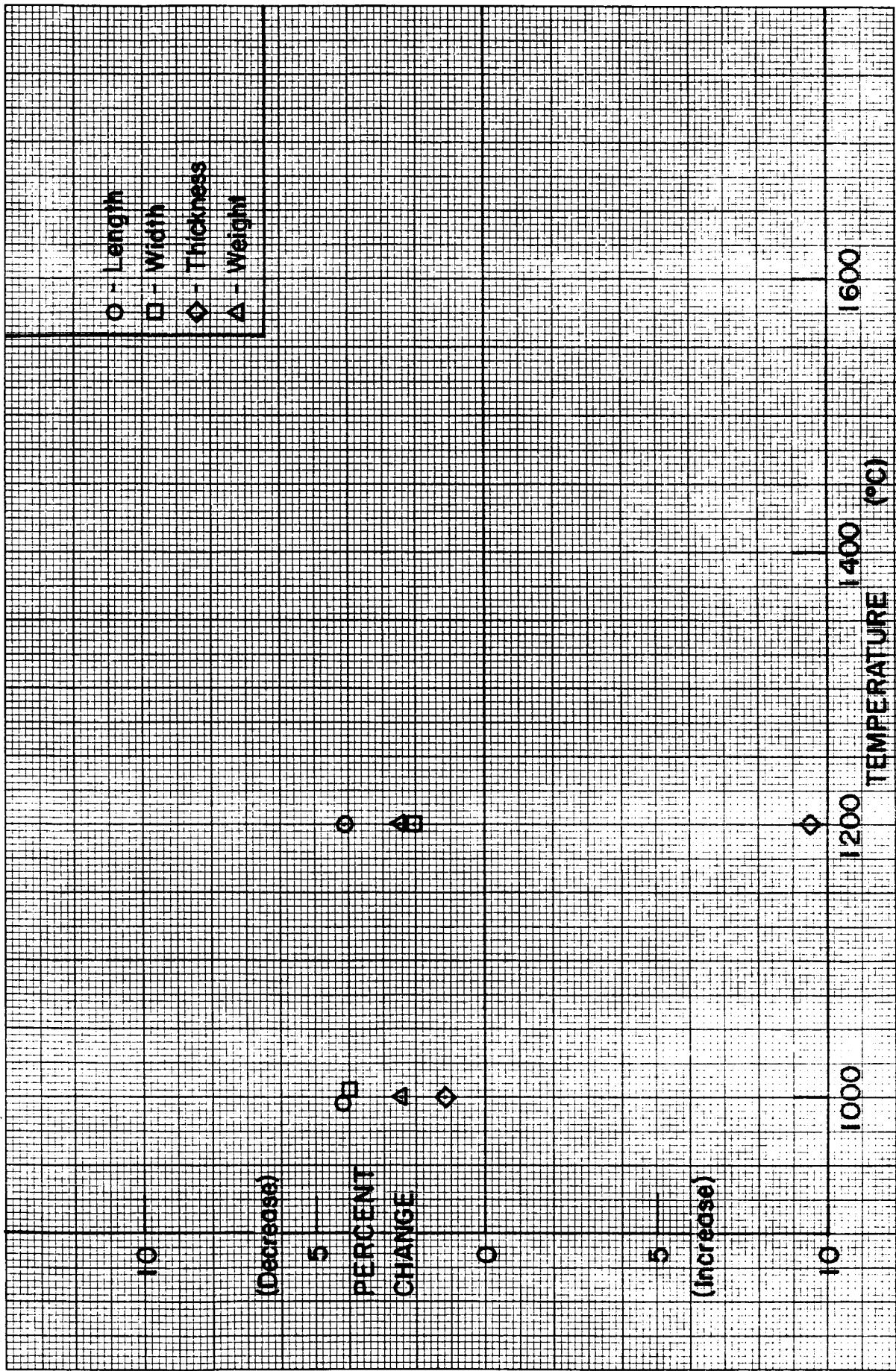


Figure 35.- Weight and dimensional changes in Irish Refrasil fiber cake after 100-hour exposure to various temperatures

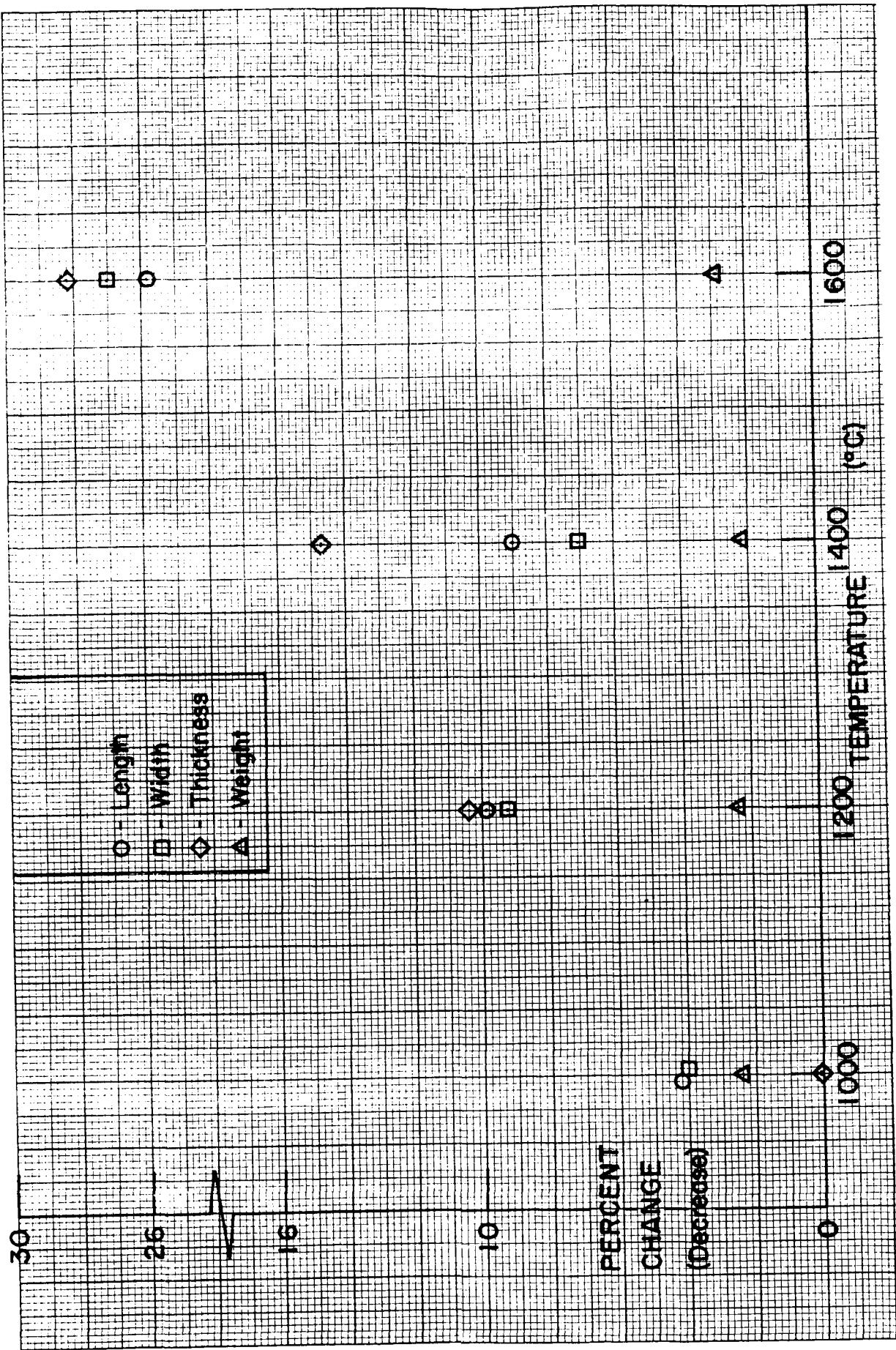


Figure 36.- Weight and dimensional changes in Saffil Alumina HT fiber cake after 100-hour exposure to various temperatures.

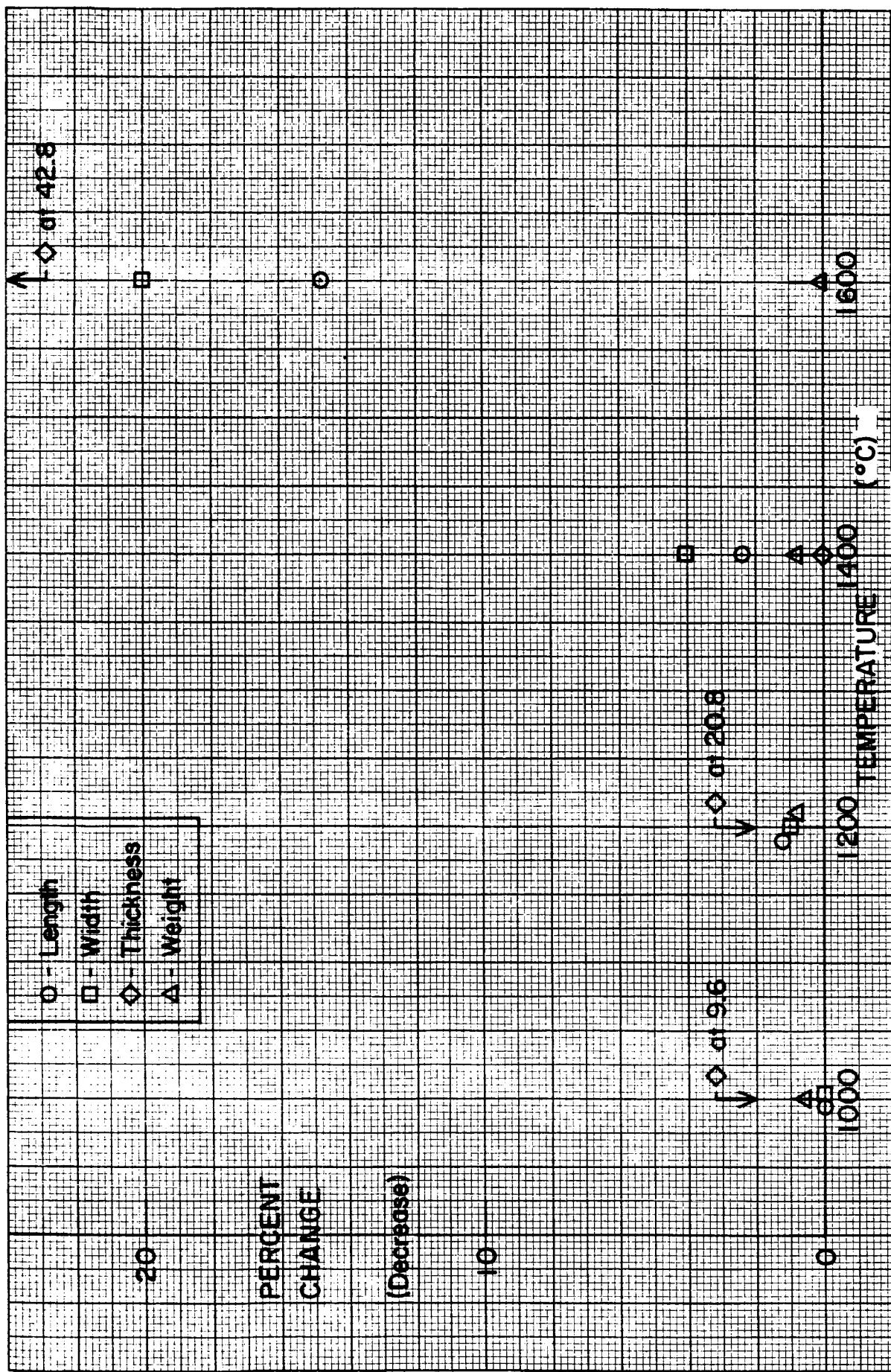


Figure 37.—Weight and dimensional changes in Saffil Zirconia HT fiber cake after 100-hour exposure to various temperatures.

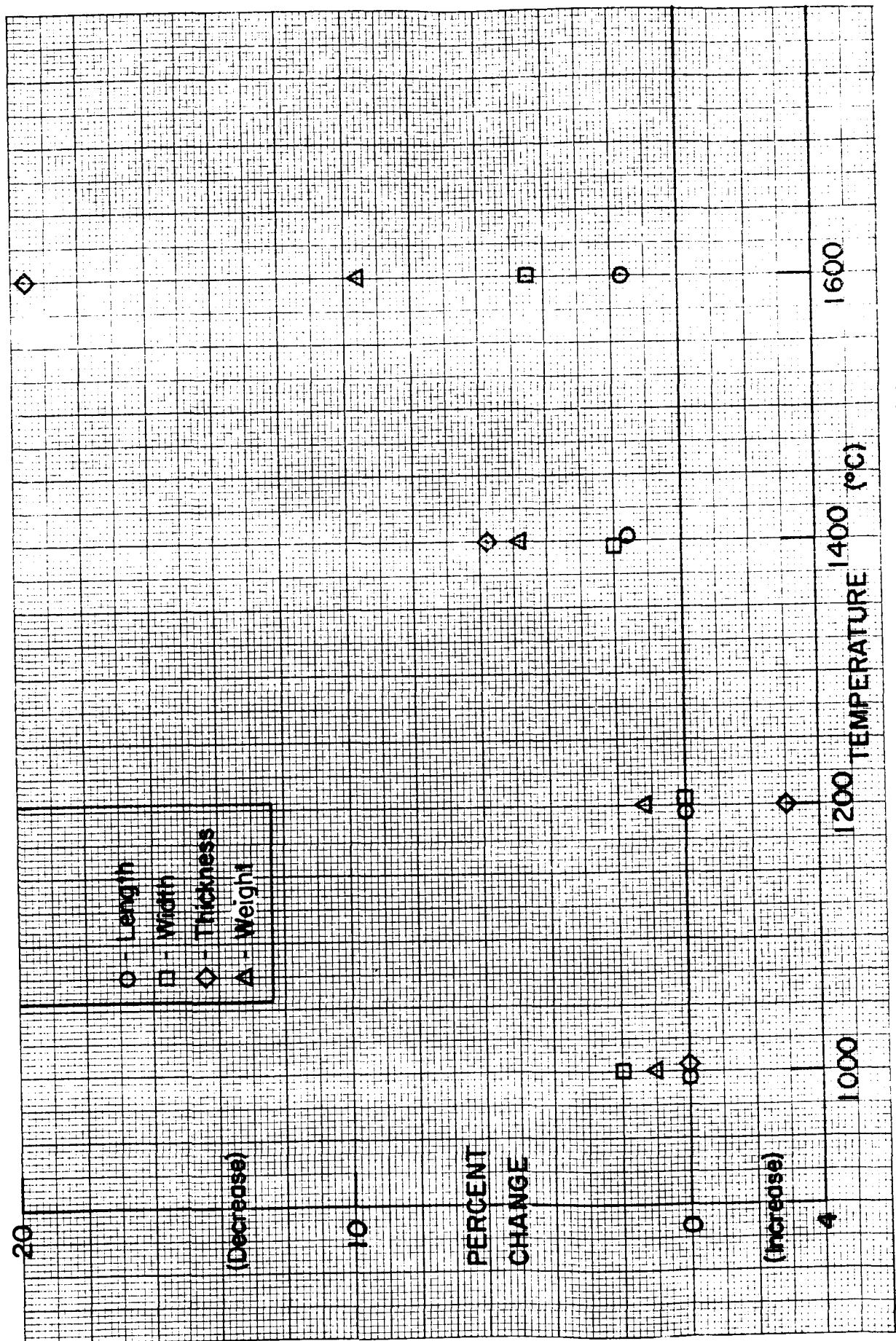


Figure 38.- Weight and dimensional changes in B&W Mullite fiber cake after 100-hour exposure to various temperatures.

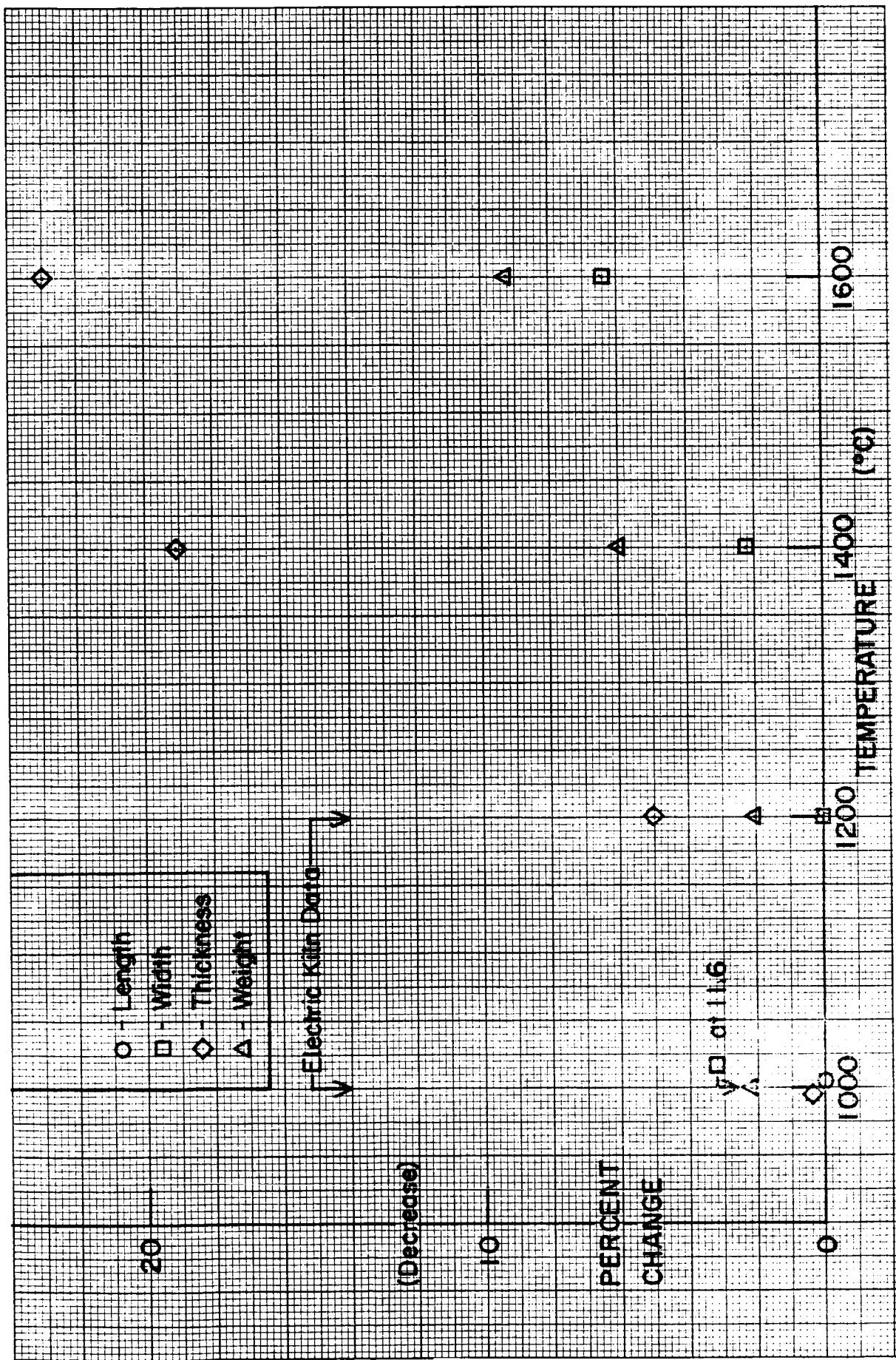


Figure 39.—Weight and dimensional changes in 3 Micron Mullite fiber cake after 100-hour exposure to various temperatures.

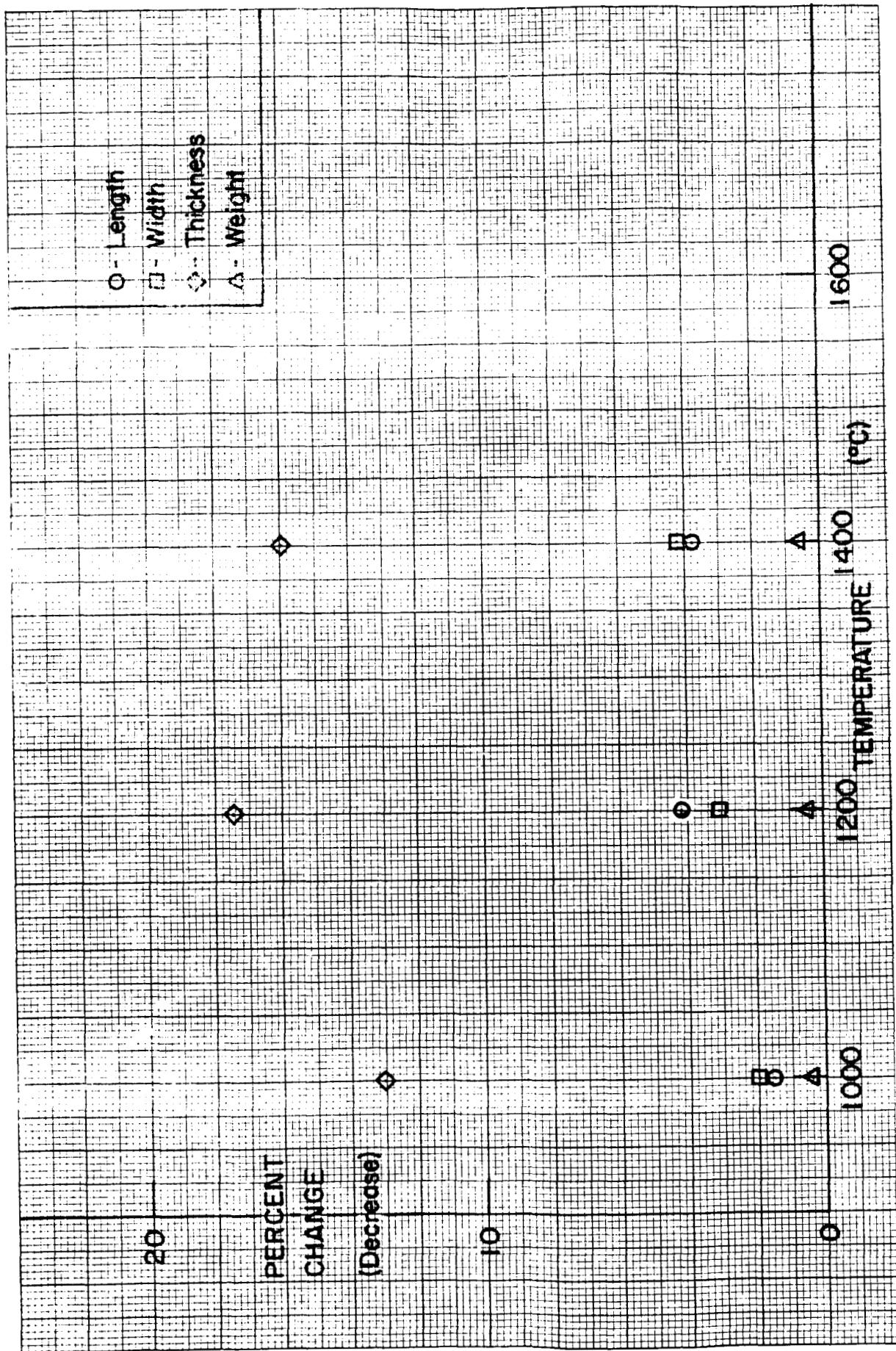


Figure 40.- Weight and dimensional changes in Fiberfrax H fiber cake after 100-hour exposure to various temperatures.

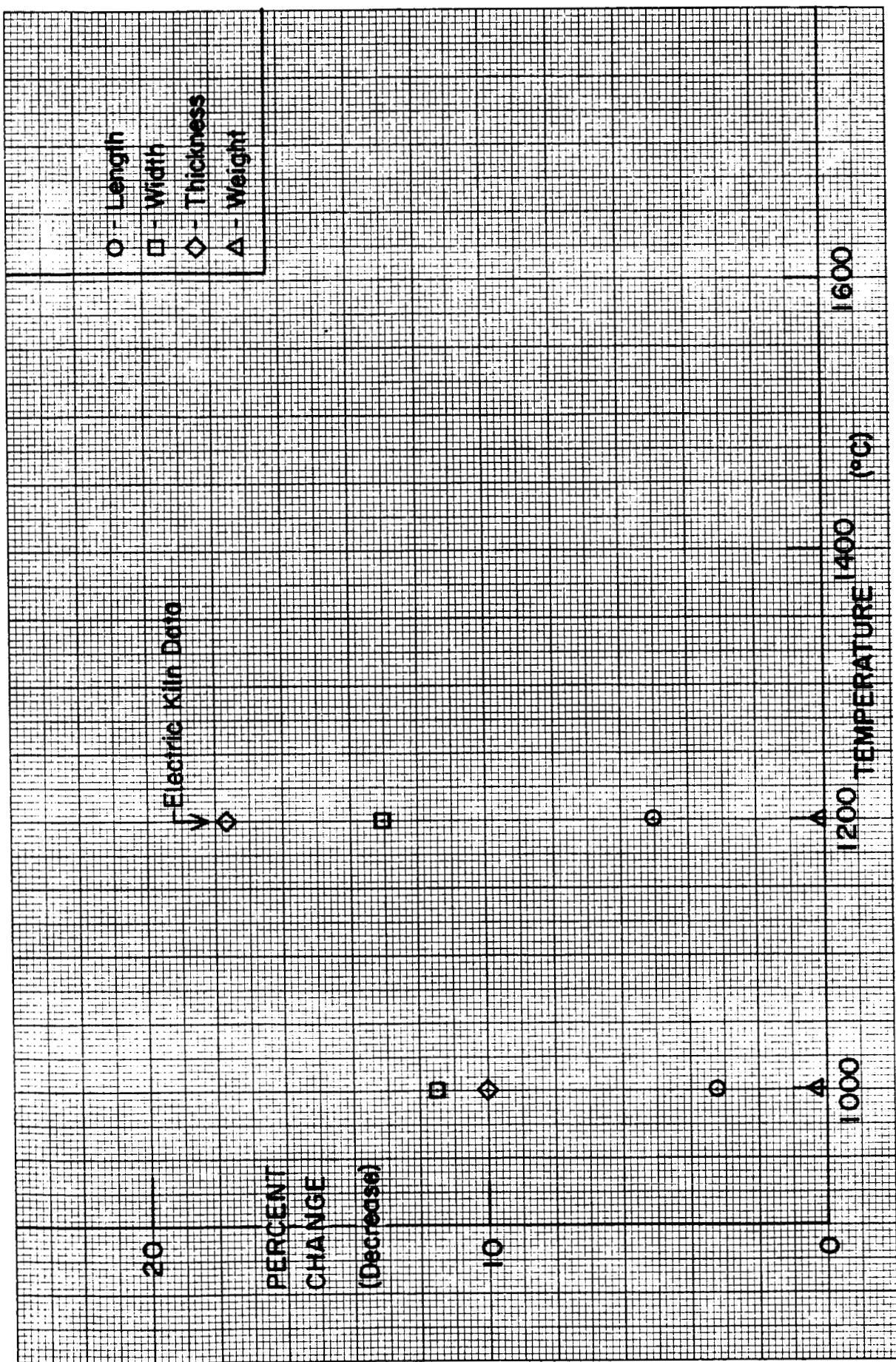


Figure 41. - Weight and dimensional changes in Fiberfrax H blanket after 100-hour exposure to various temperatures.

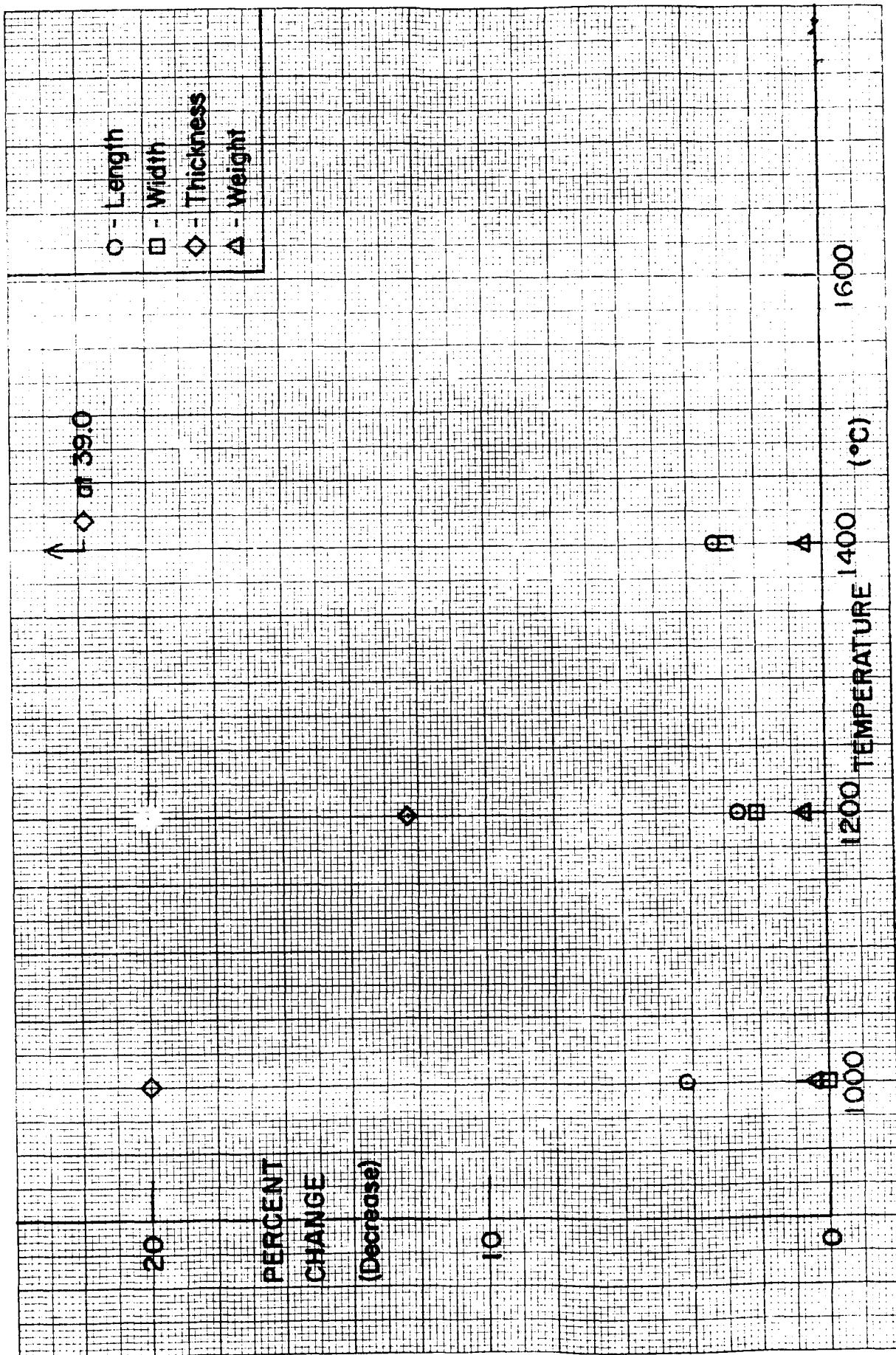


Figure 42.—Weight and dimensional changes in Kaowool blanket after 100-hour exposure to various temperatures.

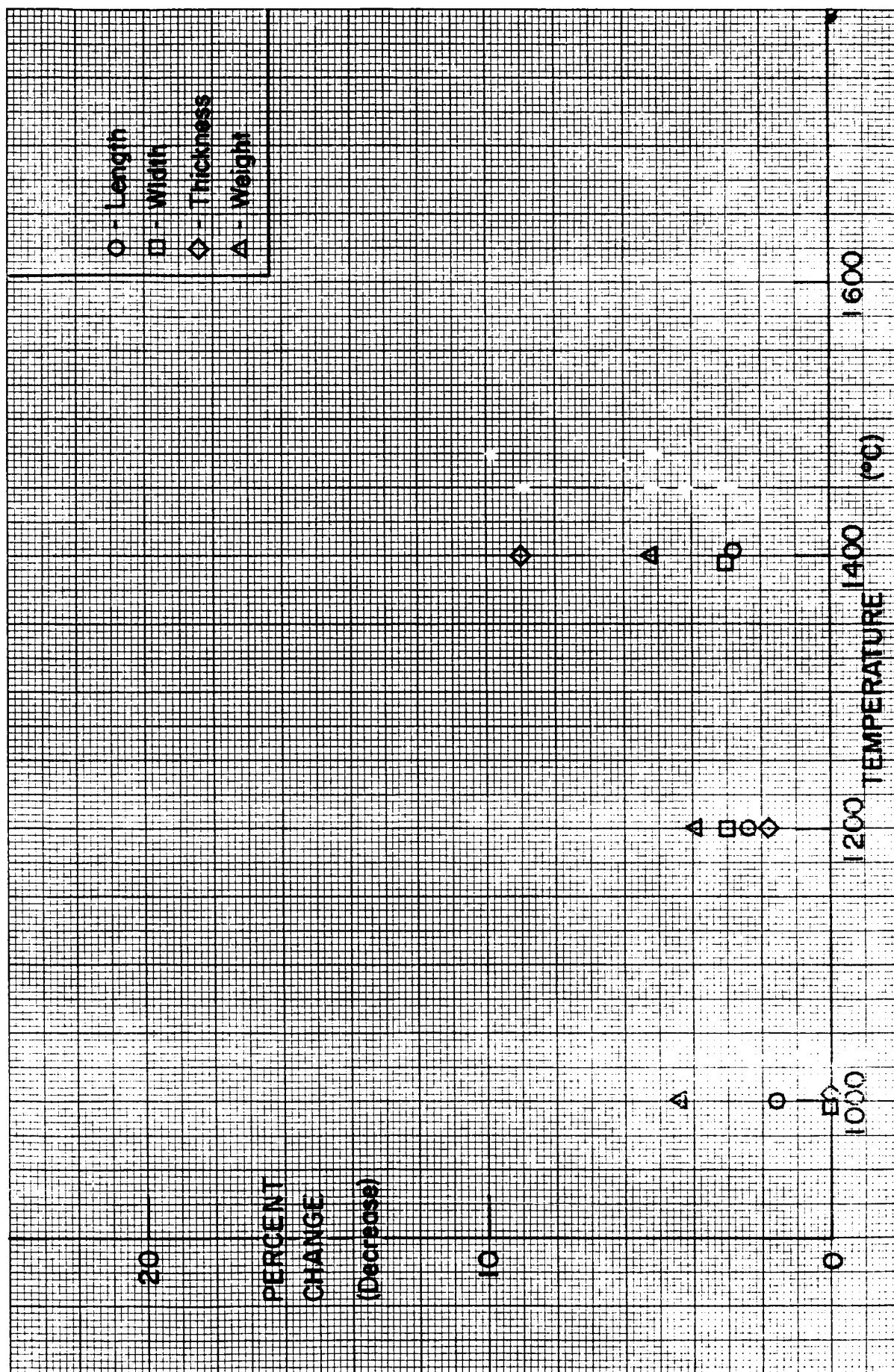


Figure 43.- Weight and dimensional changes in Fiberchrome blanket after 100-hour exposure to various temperatures.

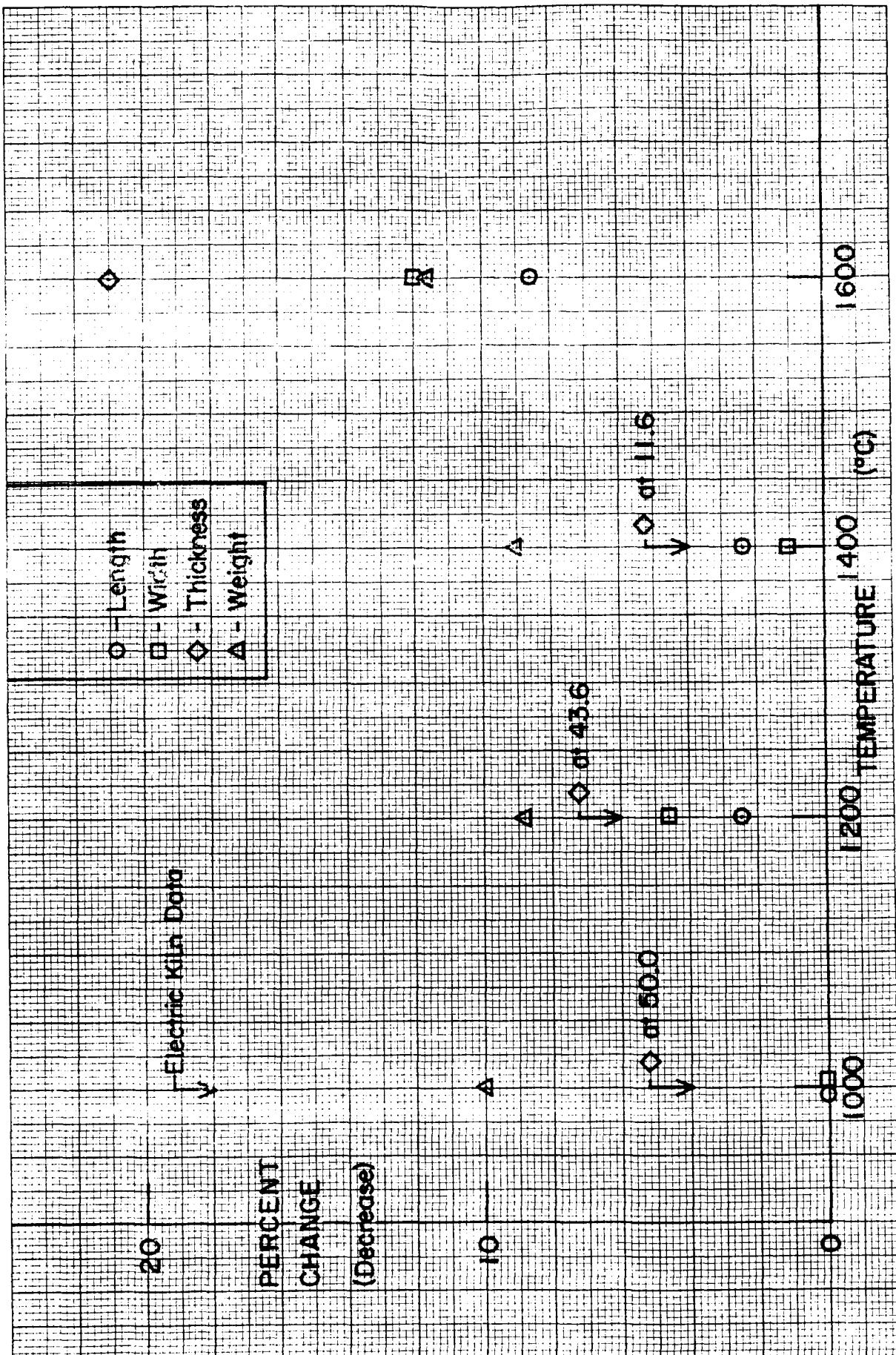


Figure 44. - Weight and dimensional changes in Saffil Alumina HT blanket after 100-hour exposure to various temperatures.

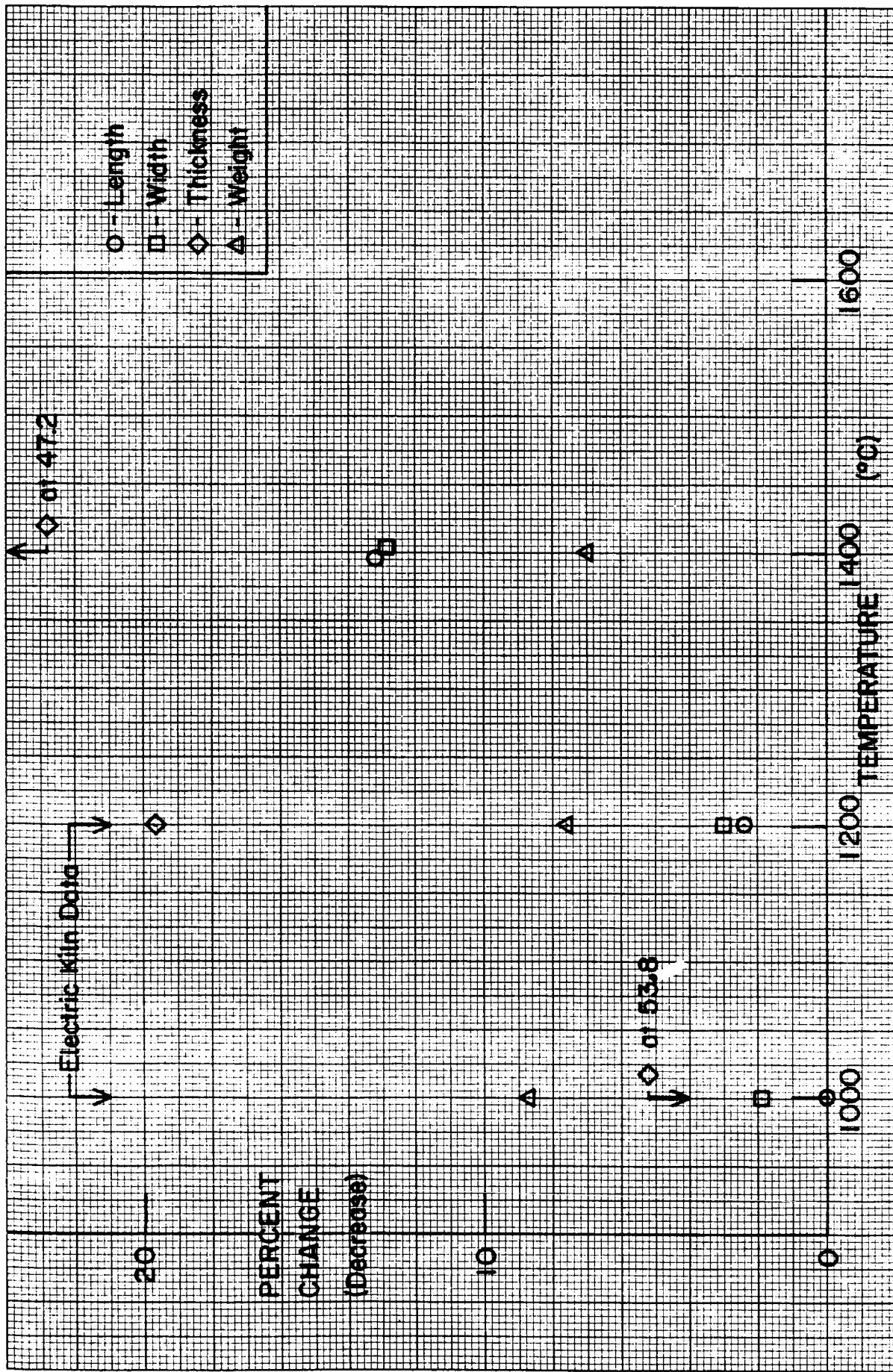


Figure 45.- Weight and dimensional changes in Saffil Zirconia HT blanket after 100-hour exposure to various temperatures.

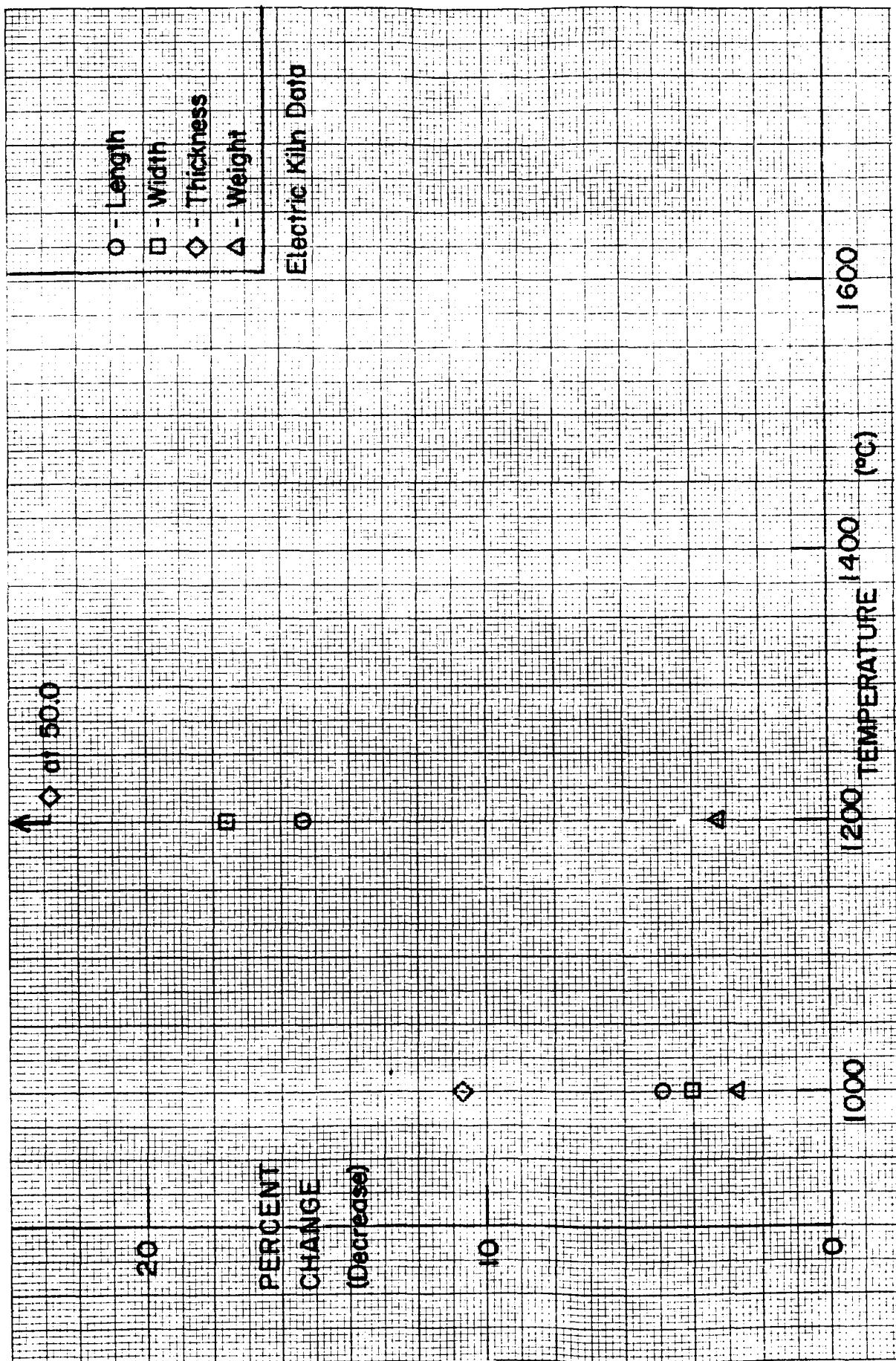
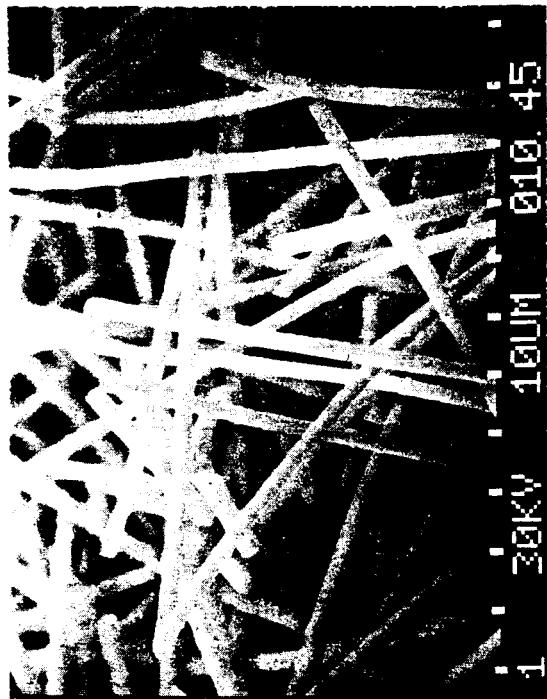
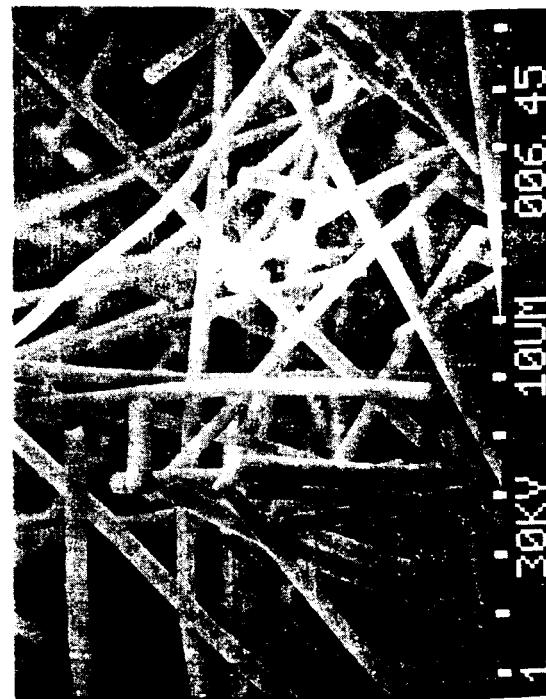


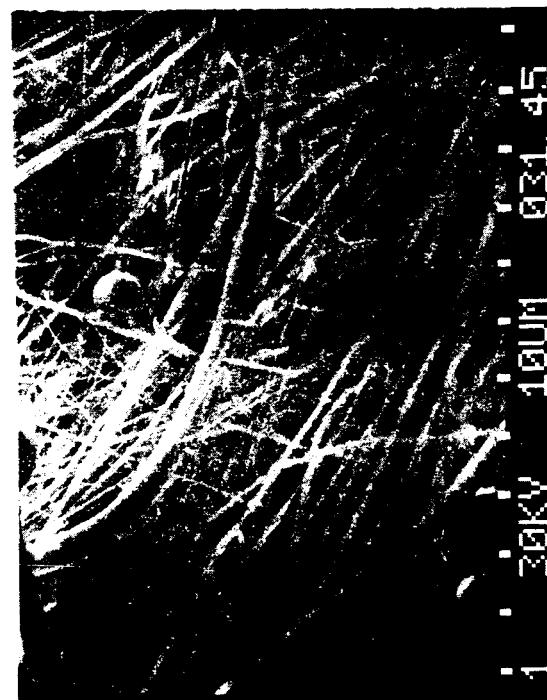
Figure 46.- Weight and dimensional changes in Microquartz blanket after 100-hour exposure to various temperatures.



(a) 500 hr at 1000°C, Irish Refrasil



(b) 100 hr at 1000°C, Irish Refrasil

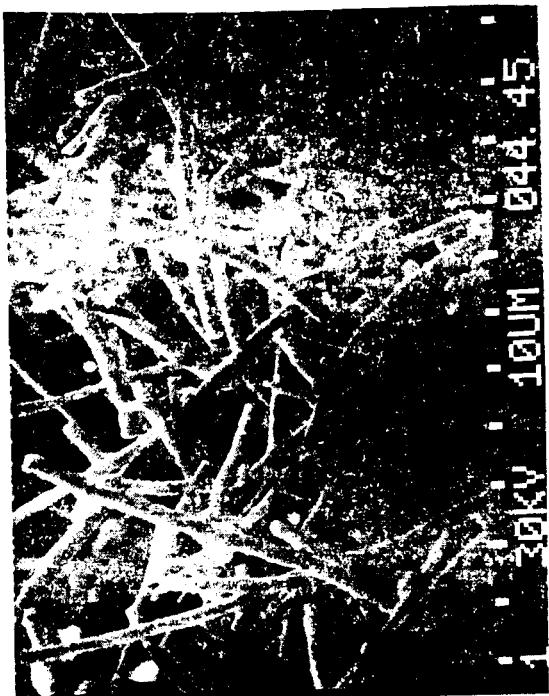


(a) 500 hr at 1000°C, Fiberfrax H



(b) 100 hr at 1200°C, Fiberfrax H

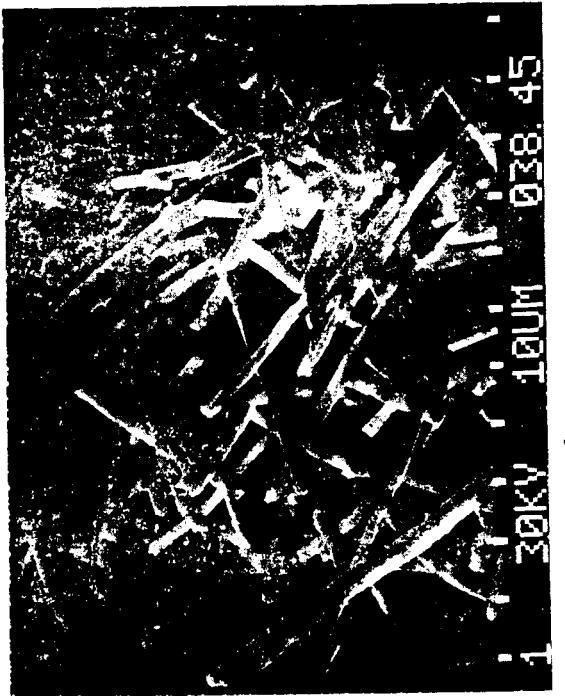
Figure 47.— SEM micrograph (1000X) of Irish Refrasil fiber cake and Fiberfrax H blanket.



(a) 500 hr at 1000°C



(b) 500 hr at 1200°C



(c) 500 hr at 1400°C

(d) 200 hr at 1600°C

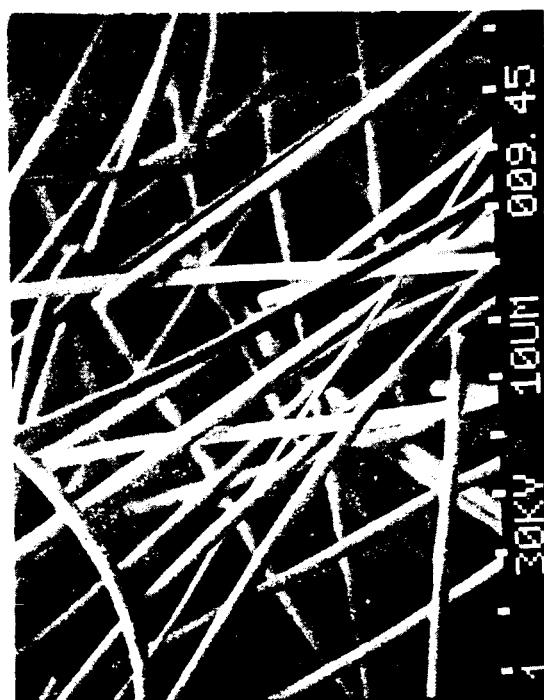
Figure 48. - SEM micrographs (1000X) of Saffil Alumina HT fiber cake.



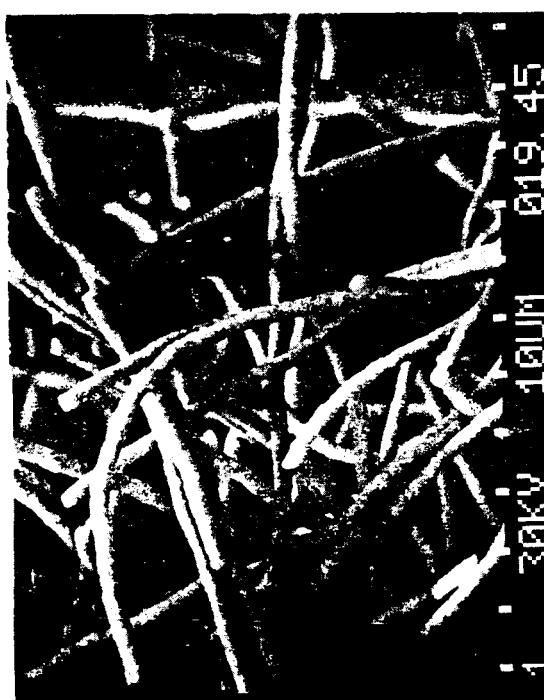
(b) 500 hr at 1200°C



(c) 500 hr at 1400°C



(a) 500 hr at 1000°C



(d) 200 hr at 1600°C

Figure 49.— SEM micrograph (10000X) of Saffil Zirconia HT fiber cake.



(a) 500 hr at 1000°C



(b) 500 hr at 1200°C

(c) 500 hr at 1400°C



(d) 200 hr at 1600°C



Figure 50.- SEM micrograph (10000X) of B&W Mullite fiber cake.

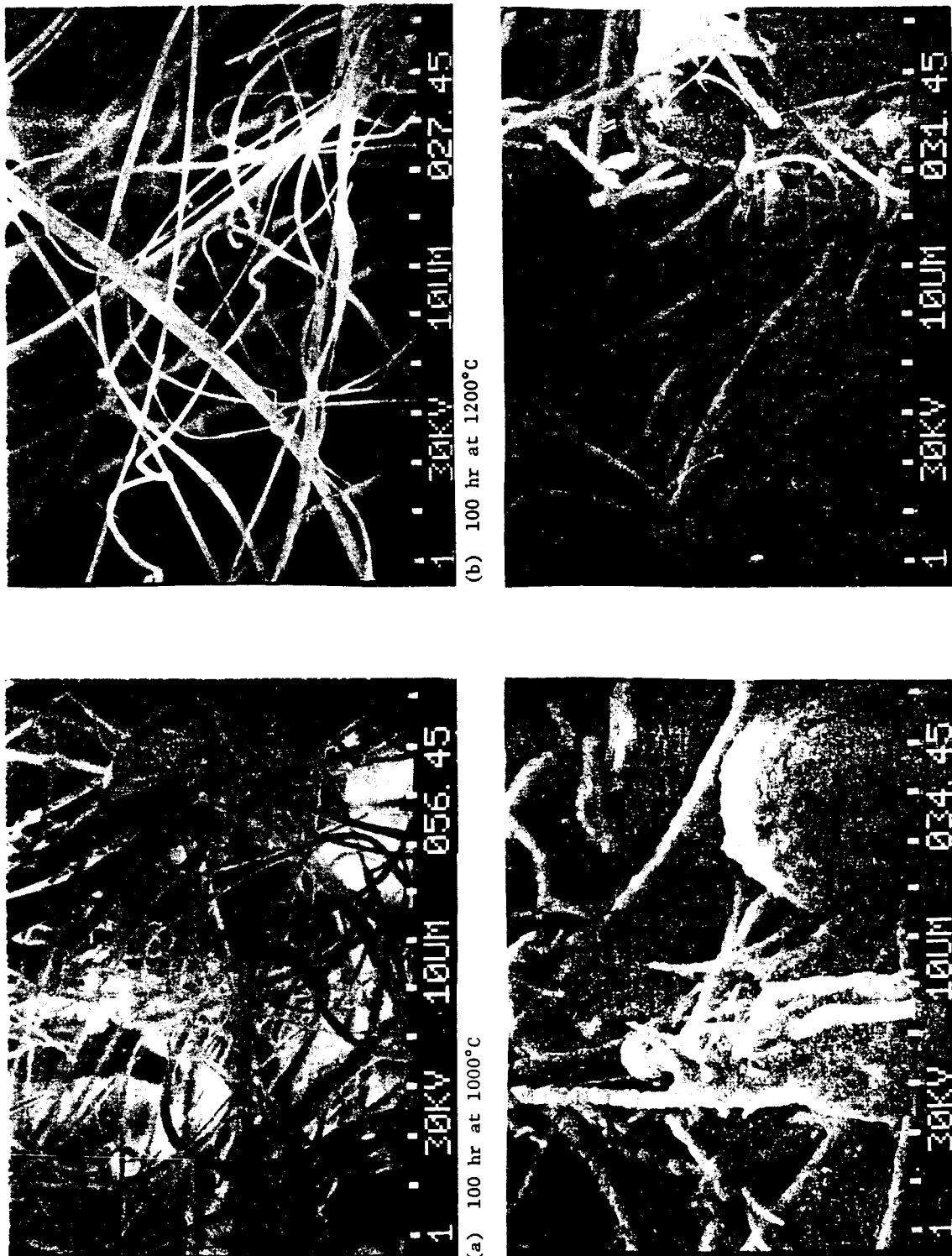


Figure 51. - SEM micrograph (1000X) of 3 micron Mullite fiber cake.



(a) 500 hr at 1000°C  
1 30kV 10μm 051.45



(b) 500 hr at 1200°C  
1 30kV 10μm 053.45

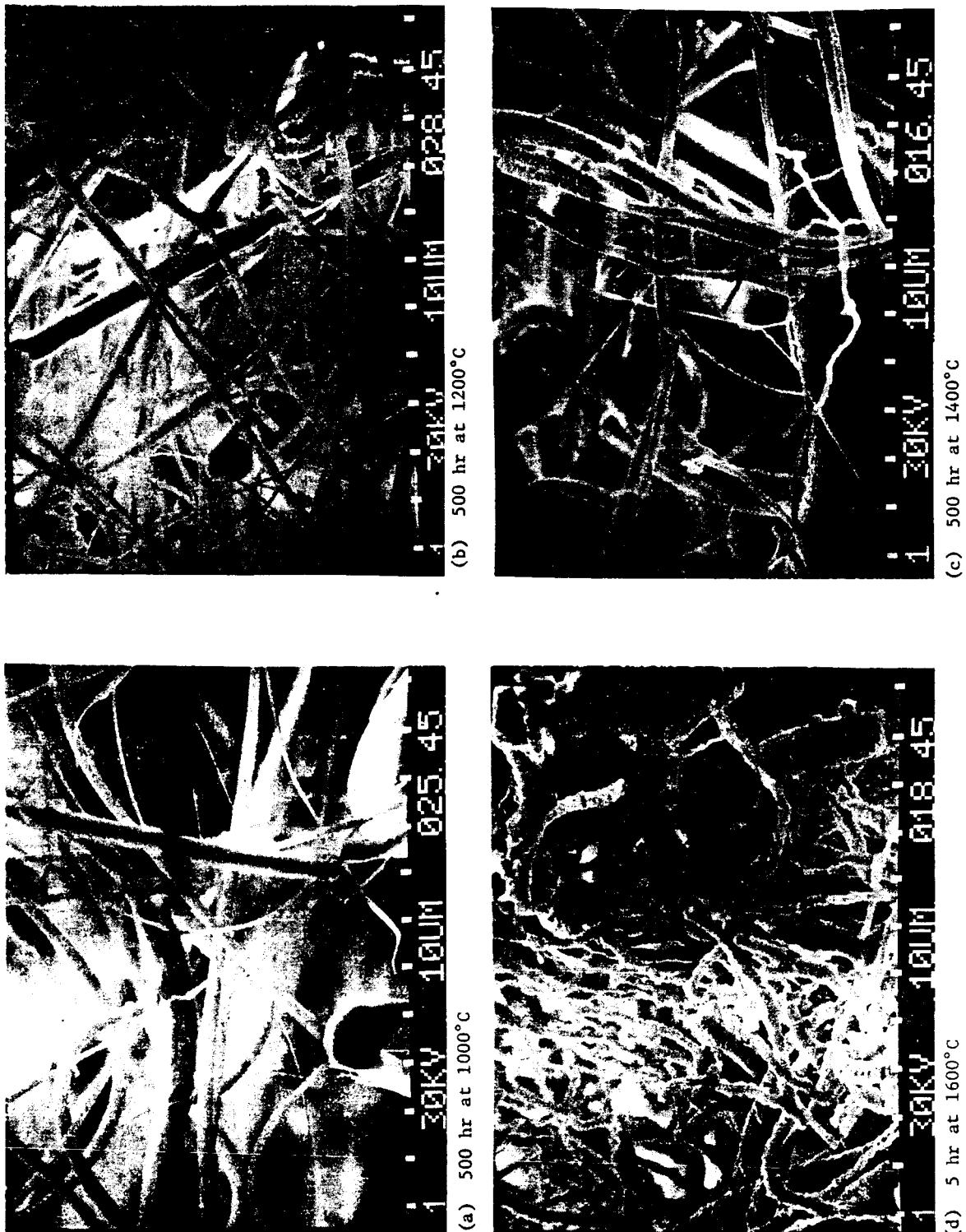


(c) 500 hr at 1400°C  
1 30kV 10μm 014.45



(d) 5 hr at 1600°C  
1 30kV 10μm 015.45

Figure 52. - SEM micrograph (1000X) of Fiberfrax H fiber cake.



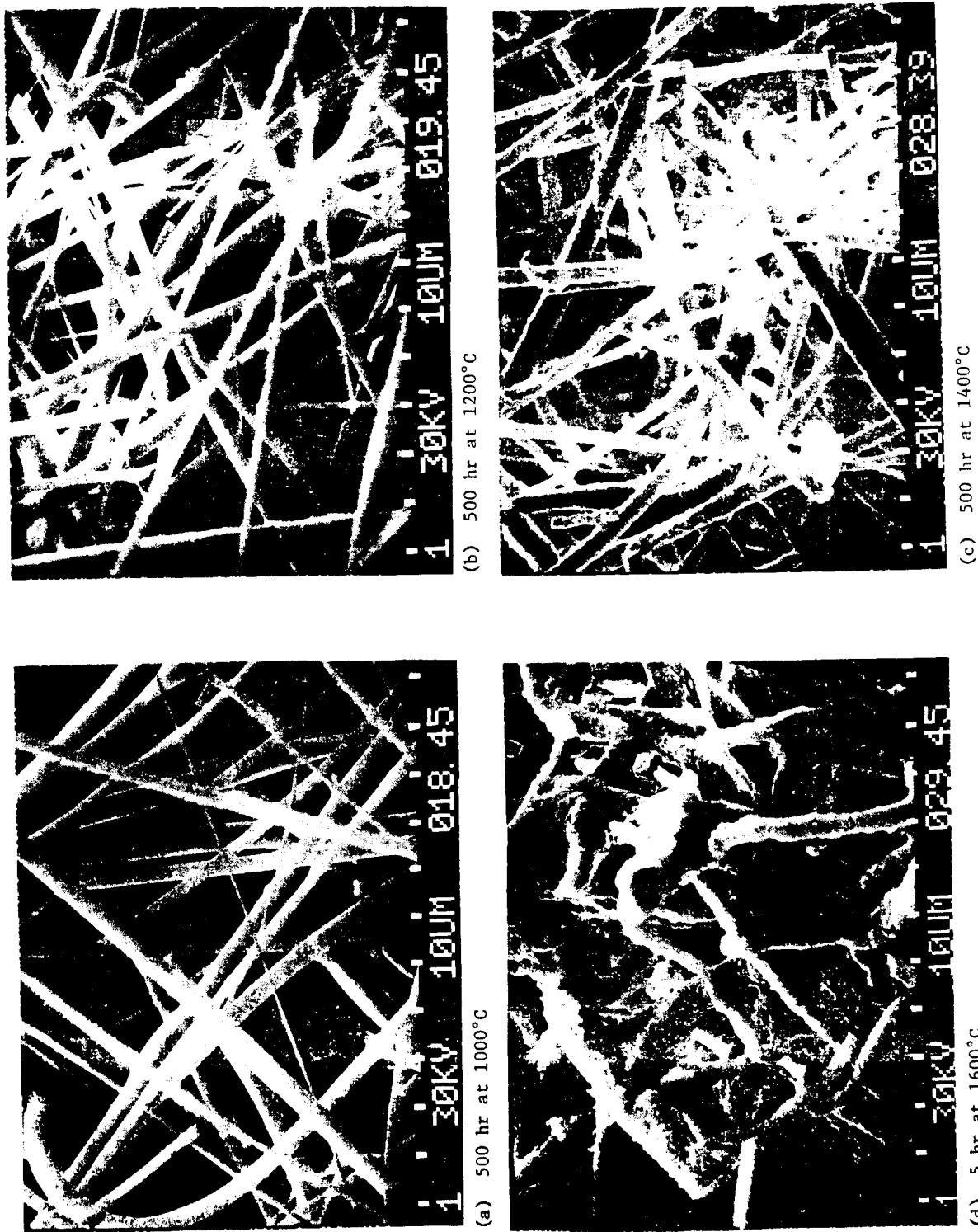


Figure 54.—SEM micrograph (1000X) of Fiberchromite blanket.

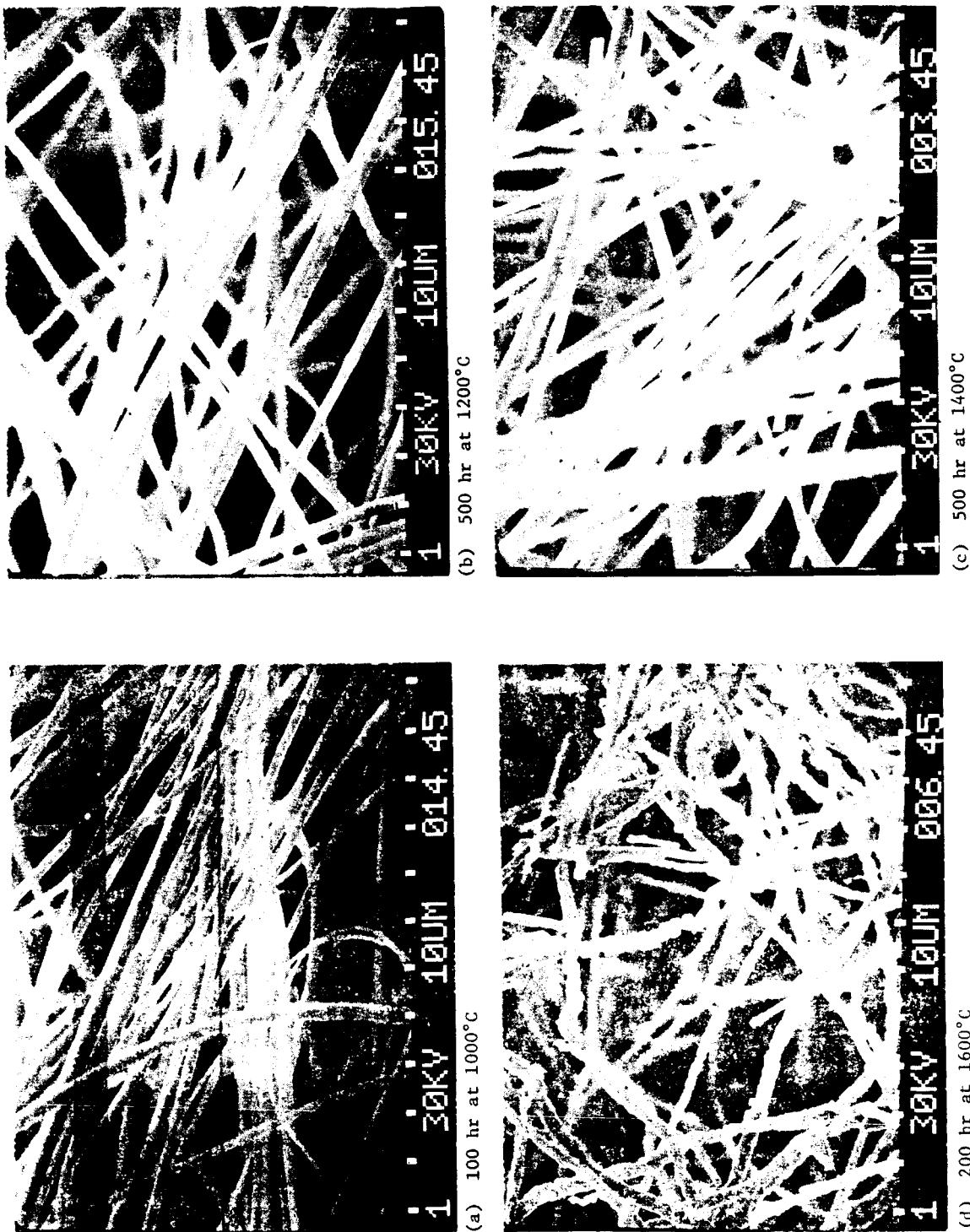


Figure 55. - SEM micrograph (1000X) of Saffil Alumina HT blanket

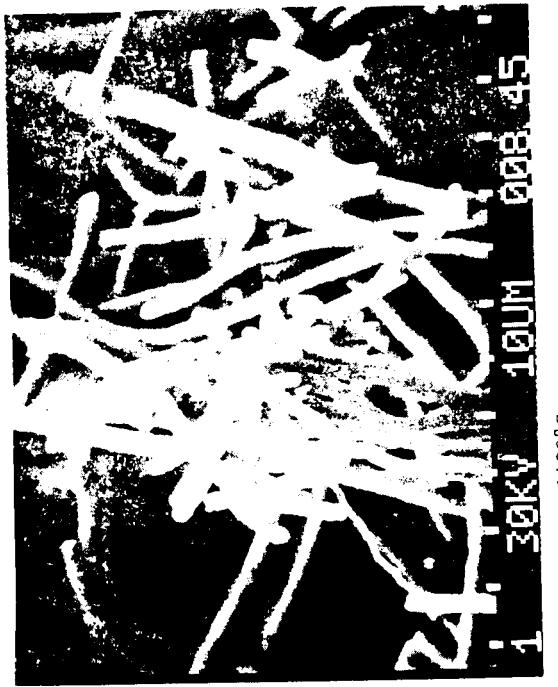
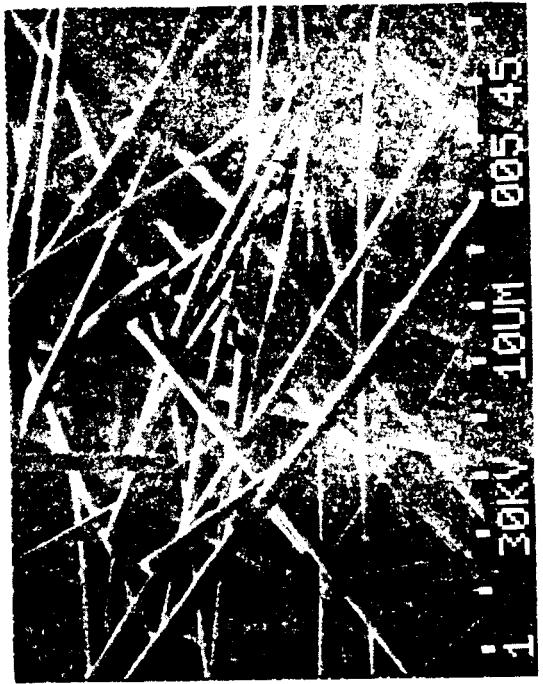
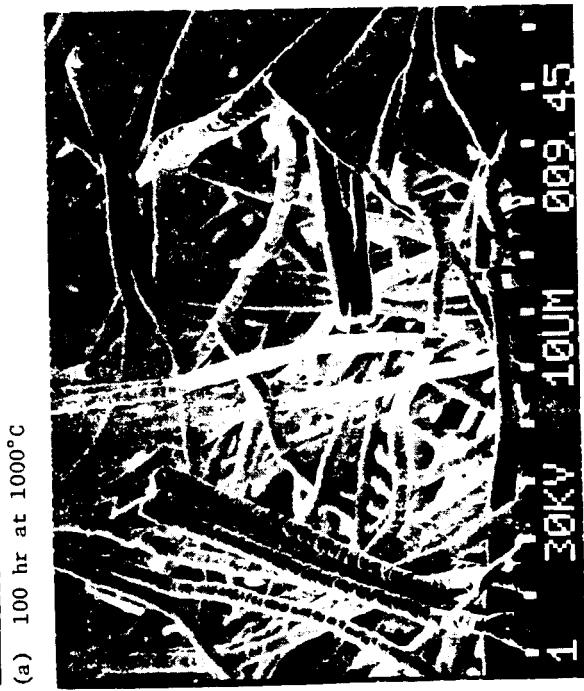
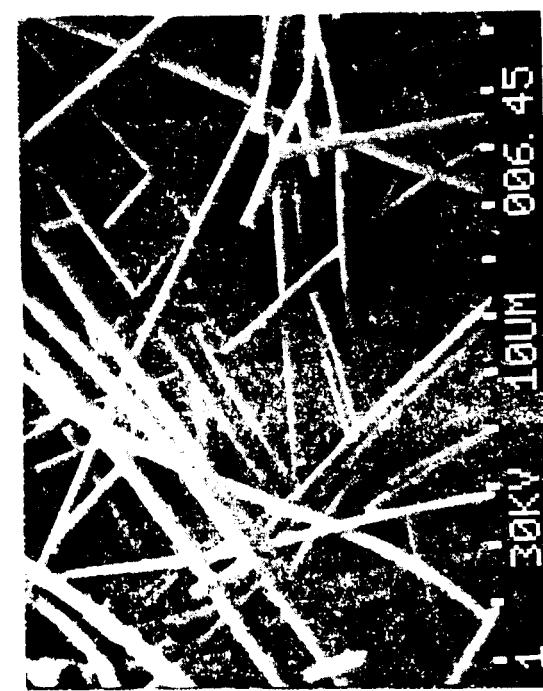


Figure 56.- SEM micrograph (1000X) of Saffil Zirconia HT blanket.

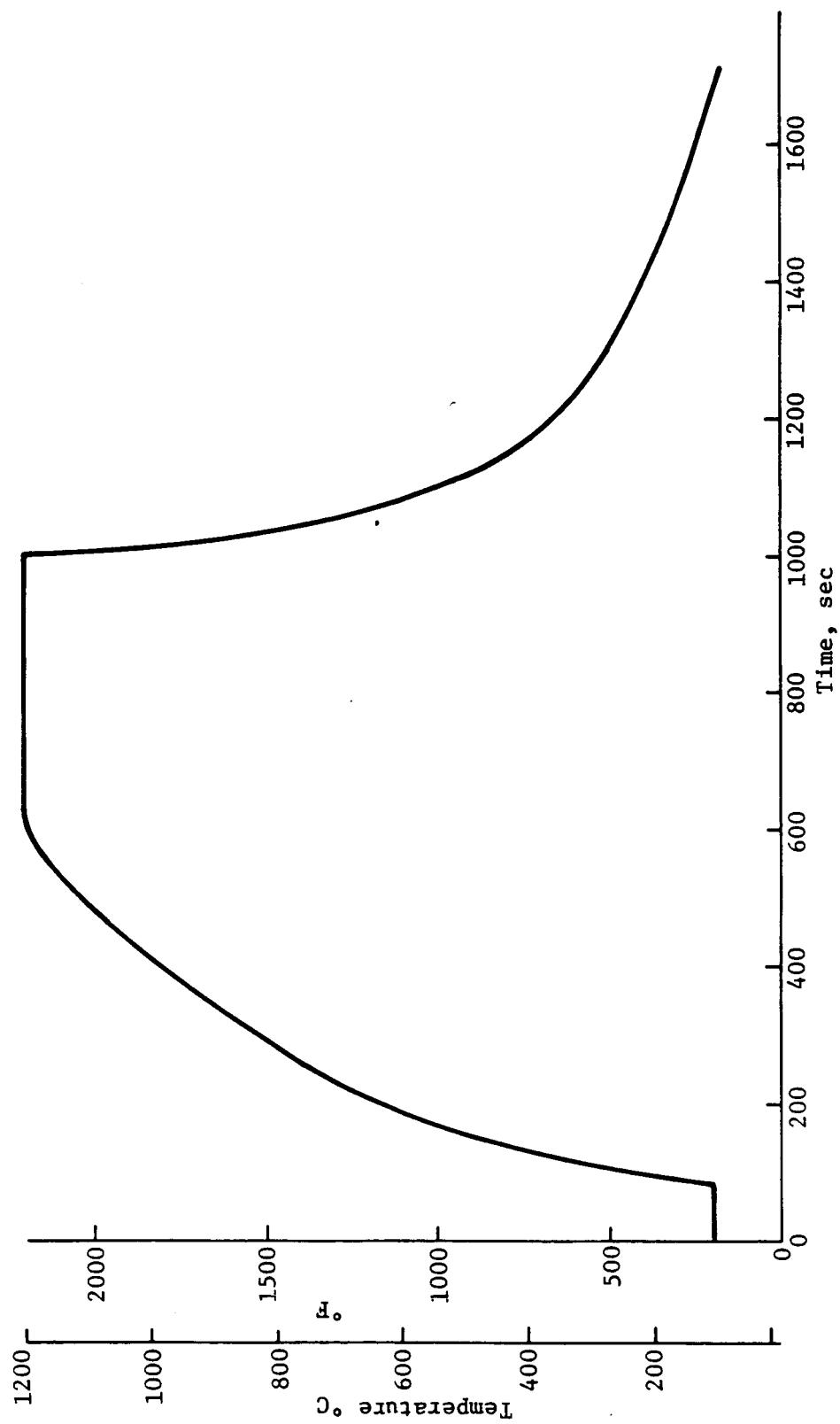
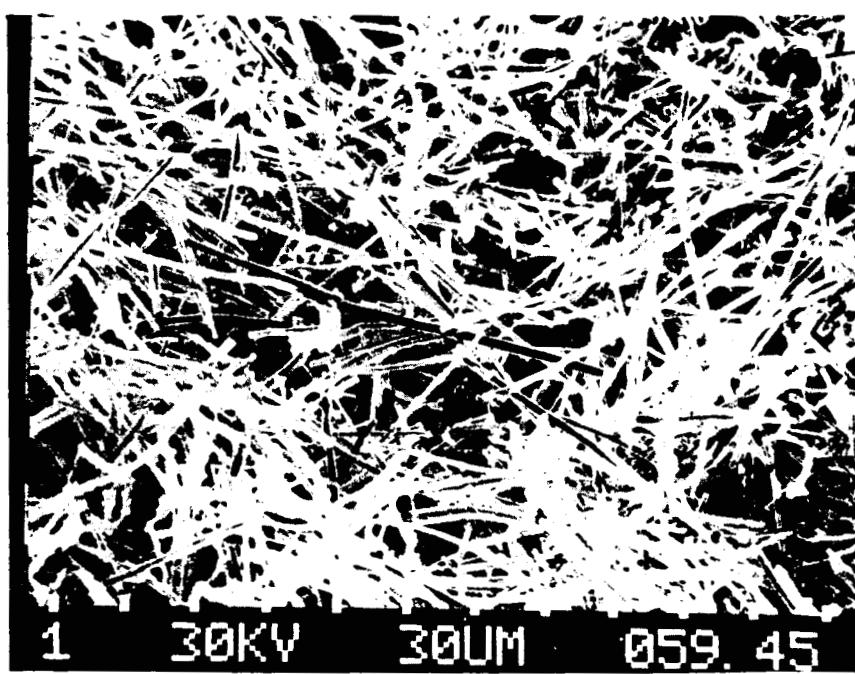


Figure 57.- 1200°C (2192°F) radiant heating cycle.



Control



25 cycles at 1400°C

Figure 58.- SEM photograph (300X) of Saffil Alumina RSI

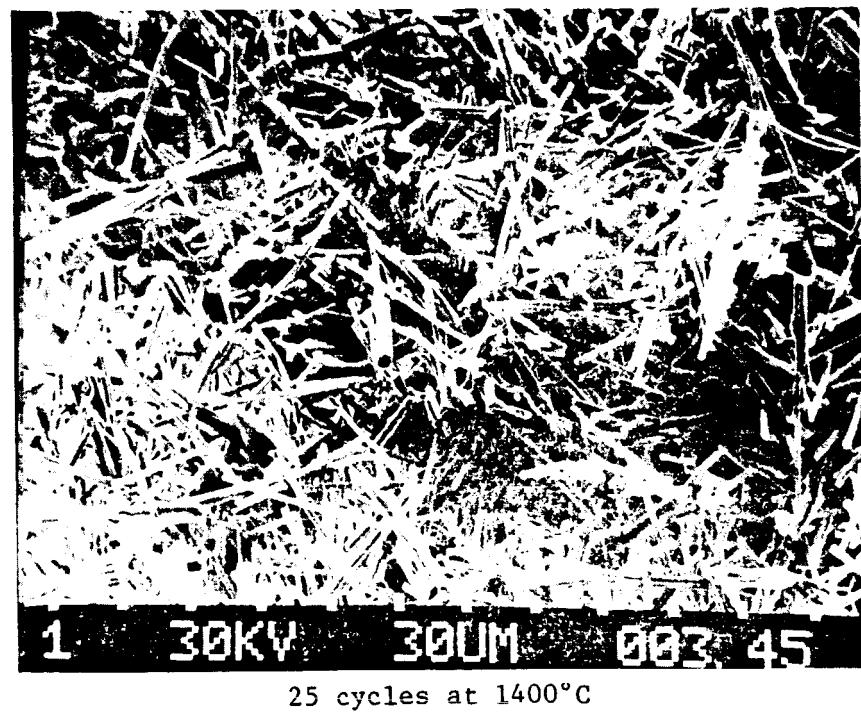
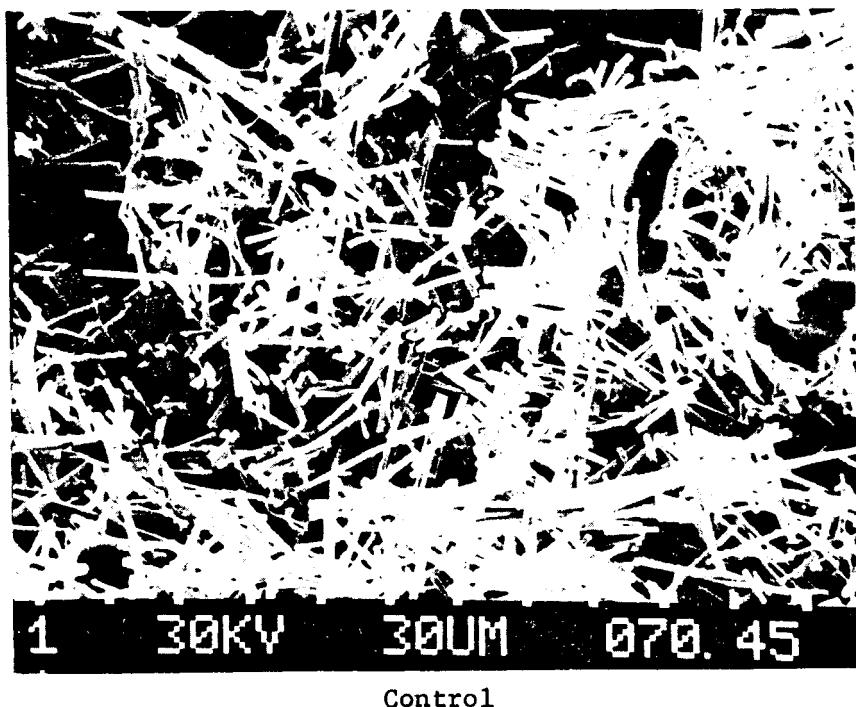
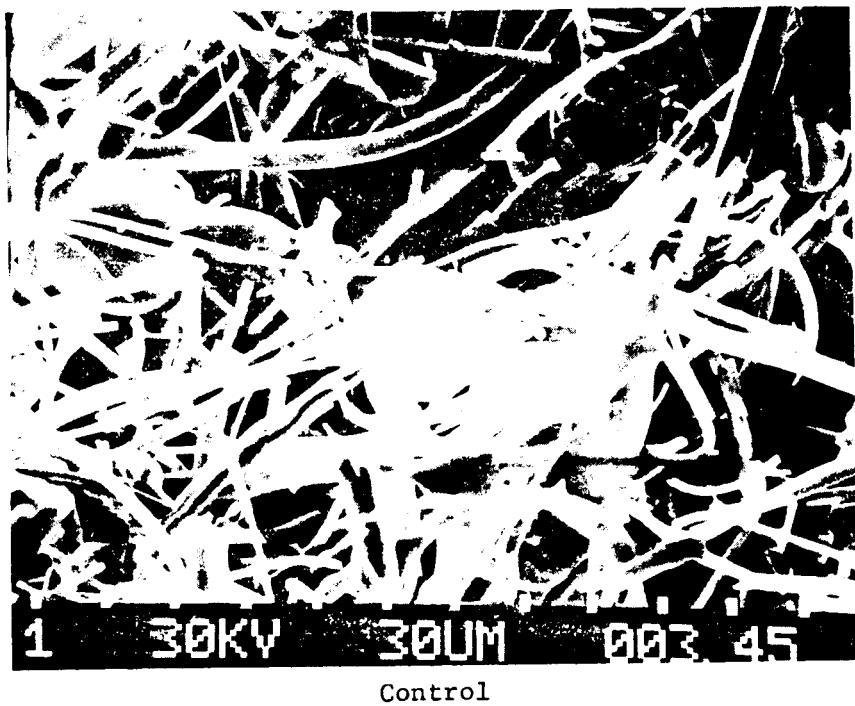


Figure 59.- SEM photograph (300X) of Saffil Zirconia RSI



Control



Figure 60.- SEM photograph (300X) of B&W Mullite RSI

**APPENDIX B**

**TABLE**

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TABLE 1.- SELECTED FIBER MATERIAL SYSTEMS

Material Designation	Material Form	Fiber Analysis*	Vendor	Price (\$/kg)
Saffil Alumina HT	Loose Fiber Wool	$\text{Al}_2\text{O}_3$ in transitional form with small percentage of other inorganic oxides added as crystal phase stabilizers	ICI United States, Inc.	20.4
Saffil Alumina HT Blanket	Blanket roll, 0.46 m (1.5 ft) wide x 1.59 cm (5/8-in.) thick; .072 g/cc (4.5 lb/ft <sup>3</sup> ) density.		ICI United States, Inc.	23.7
Saffil Zirconia HT	Loose Fiber Wool	$\text{ZrO}_2$ with small percentage of rare earth oxides added as crystal phase stabilizers	ICI United States, Inc.	22.6
Saffil Zirconia HT Blanket	Blanket roll, 0.46 m (1.5 ft) wide x 1.27 cm (0.5-in.) thick; .072 g/cc (4.5 lb/ft <sup>3</sup> ) density.		ICI United States, Inc.	25.9
Fiberfrax H Fiber	Bulk Fiber	62% $\text{Al}_2\text{O}_3$ ; 38% $\text{SiO}_2$	Carborundum	5.1
Fiberfrax H Blanket	Blanket roll, 0.61 m (2 ft) wide x 2.54 cm (1 in.) thick; .096 g/cc (6 lb/ft <sup>3</sup> ) density.		Carborundum	5.5
Mullite Fiber	Bulk Fiber	77±1% $\text{Al}_2\text{O}_3$ ; 17±1% $\text{SiO}_2$ ; 4.5±.5% $\text{B}_2\text{O}_3$ ; 1.5±.2% $\text{P}_2\text{O}_5$ ; Less than 0.5% trace impurities.	Babcock and Wilcox	264.6
3 Micron Mullite Fiber	Bulk Fiber	96.6% $\text{SiO}_2$ ; 3.4% $\text{Cr}_2\text{O}_3$	Babcock and Wilcox	+ Hitco
Irish Refrassil Fiber, Type SS-19-A5	Bulk Fiber			66.2
Kaowool 1400 Blanket	Blanket roll, 0.31 m (1 ft) wide x 1.27 cm (0.5 in.) thick; .128 g/cc (8 lb/ft <sup>3</sup> ) density.	38.6% $\text{SiO}_2$ ; 60.82% $\text{Al}_2\text{O}_3$ ; 0.58% impurities.	Babcock and Wilcox	17.9
Fiberchrome Felt	Blanket sheet, 1.07 m (3.5 ft) wide x 1.22 m (4 ft) long x 1.27 cm (0.5 in.) thick; .128 g/cc (8 lb/ft <sup>3</sup> ) density.	55% $\text{SiO}_2$ ; 40.5% $\text{Al}_2\text{O}_3$ ; 4% $\text{Cr}_2\text{O}_3$ ; 0.21% FeO; 0.15% $\text{Na}_2\text{O}$	Johns-Manville	10.5
Microquartz Felt	Blanket sheet, 0.92 m (3 ft) wide x 1.53 m (5 ft) long x 1.27 cm (0.5 in.) thick; 0.96 g/cc (6 lb/ft <sup>3</sup> ) density.	98.50% $\text{SiO}_2$ ; 0.1% max. B; 0.06% max. Fe; 0.50% max. $\text{Al}_2\text{O}_3$ ; 0.35% max. $\text{CaO}$ ; 0.35% max. $\text{MgO}$ ; 0.15% max. $\text{Na}_2\text{O}$	Johns-Manville	18.1

\*Data from References 1 through 6.

+Material supplied by NASA Lewis Research Center.

TABLE 2.- FIBER DIAMETER AND SHOT CONTENT

Fiber	Mean Fiber Dia. ( $\mu$ )	Diameter Range ( $\mu$ )	Shot Content	Shot Concentration (No. per sq. cm)
Irish Refrasil	3.05	2.5 - 3.5	None observed	-----
Fiberfrax H	2.15	0.2 - 6.0	15 pcs of shot in area 400 x 300 $\mu$	12,500
Saffil Alumina HT	2.08	1.5 - 3.5	None Observed	-----
Saffil Zirconia HT	2.76	2.0 - 4.0	Single piece of shot in area 870 x 1200 $\mu$	100
B&W Mullite	3.22	0.8 - 9.0	10 pieces of shot in area 400 x 300 $\mu$	8,300
3 Micron Mullite	2.00	0.5 - 5.0	Portions of 7 pieces of Shot in area 110 x 80 $\mu$	40,000

TABLE 3.-TIME AND TEMPERATURE EXPOSURES OF VARIOUS MATERIALS

Fiber	Material Type	1000°C				1200°C				1400°C				1600°C			
		Hours		Hours		Hours		Hours		Hours		Hours		Hours			
		25	100	500	25	100	500	25	100	500	25	100	500	25	100	200	
Irish Refrasil	Felted Fiber Cake	G	G	G	G	G	G	--	--	--	--	--	--	--	--	--	
Saffil Alumina HT	Felted Fiber Cake	G	G	G	G	G	G	GE	G	G	G	G	G	G	G	G	
Saffil Zirconia HT	Felted Fiber Cake	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	
B&W Mullite	Felted Fiber Cake	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	
3 Micron Mullite	Felted Fiber Cake	E	E	--	E	E	--	G	G*	G*	G	G	G	G	G	G	
Fiberfrax H	Felted Fiber Cake	G	G	G	G	G	G	G	G	G	G	G	G	G	G	G	
Fiberfrax H	Blanket	GE	GE	G	GE	GE	--	--	--	--	--	--	--	--	--	--	
Kaowool 1400	Blanket	GE	GE	G	GE	GE	--	--	--	--	--	--	--	--	--	--	
Fiberchrome	Blanket	GE	GE	G	G	G	G	G	G	G	G	G	G	G	G	G	
Saffil Alumina HT	Blanket	E	E	--	G	G	G	GE	G	G	G	G	G	G	G	G	
Saffil Zirconia HT	Blanket	E	E	--	GE	E	G***	GE	G	G	G	G	G	G	G	G	
Microquartz	Blanket	GE	GE	--	E	E	--	--	--	--	--	--	--	--	--	--	

NOTES: G and E indicate exposure conditions for times shown

G = Gas Furnace; E = Electric Furnace; -- = No Exposure

\* 75 and 475 hour exposure respectively

\*\* 5 hour exposure

\*\*\* 425 hour exposure

TABLE 4.—VISUAL APPEARANCE OF INSULATION MATERIALS AFTER 1000°C (1832°F) EXPOSURE IN GAS-FIRED KILN

Visual Observation After 1000°C (1832°F) Exposure		500 Hours	
Fiber	Material Type	25 Hours	200 Hours
Felted Fiber	Irish Refrassil Cake	Loss of green color. Green bled onto setter plate.	Upper surface almost white. Further leaching of chroma from fiber cake.
Felted Fiber	Saffil Alumina Cake	No visual change.	No visual change.
Felted Fiber	Saffil Zirconia Cake	2-1/2 x 1-1/4 samples puffed up to 1-1/4 in. height.	Delaminations appearing in samples. 2-1/2 x 1-1/4 samples puffed up to 1-1/8 in. height.
Felted Fiber	BW Mullite Cake	No visual change.	No visual change.
Fiberfrax H	Blanket	No visual change.	No visual change.
Fiberfrax H	Kaowool	Corners turned up. Crusty surface.	Material has hardened and shrank in length.
Microquartz	Blanket	Thickness has shrunk to 5/16 in. Specimens are bowed.	Thickness has shrunk to 1/2 in. Surface is hard and crusty.
Fiberglass	Blanket	Color change from grey to greenish tint.	Same as after 5 hours.

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TABLE 5.—VISUAL APPEARANCE OF INSULATION MATERIALS AFTER 1200°C (2192°F) EXPOSURE IN GAS-FIRED KILN

Fiber	Material Type	Visual Observation After 1200°C (2192°F) Exposure			350 Hours	500 Hours
		25 Hours	100 Hours	200 Hours		
Irish Refrasiil	Felted Fiber Cake	Specimen edges have turned white. Green color transferred to setter plate.	Further loss of green color. Surface turning light brown.	Removed from furnace run after 100 hours.	---	---
Saffil Alumina	Felted Fiber Cake	No apparent change.	Large shrinkage in Specimen A. Chalky surface appearance. Edge adjacent to Refrasiil has picked up trace of green contamination.	Rigid. Slight bowing in specimens. Retains white color.	Rigid and chalky surface. Slight bowing. Sample A - large shrinkage; B - moderate shrinkage; C - intermediate shrinkage after 150 hours.	Board-like consistency. Pink color-possible contamination from Fiberchrome. Chalky texture.
Saffil Zirconia	Felted Fiber Cake	Specimens are puffed up in thickness. Not sticking to setter plate.	Specimens puffed up to non-uniform thickness. Bottom surface more rigid with crusty texture.	Puffed up and uneven. Bottom rigid and top soft.	Puffed up to uneven height. Semi-rigid. No other changes.	Pigid and stiff. No other changes. Puffed up. White color.
BM Mullite	Felted Fiber Cake	Specimen texture is softer.	Same as after 5 hours.	Soft but holds together well. Retains white color.	Same as after 200 hrs.	Soft and flexible. White. Flat and square. Same as after 200 hrs.
Fiberfrax H	Felted Fiber Cake	Surface remains dimpled as after felting. No change.	Same as after 5 hours.	Sample slightly twisted. Retains white color.	Same as after 200 hrs.	Same as after 200 hrs.
92 Fiberfrax H	Blanket	Distorted shape with crusty surface. Curled at edges.	Specimens severely twisted.	Removed from furnace run after 100 hours.	---	---
Kaowool	Blanket	Thickness shrunk to about 1.1 cm (7/16 in.). No other changes.	Same as after 5 hours.	Samples are thin but remain flat and square and retain white color.	Remains flexible. Slight curling of corners. No crust formed. White color.	Semi-flexible. Edges deformed. Slight sticking to setter plate.
Fiberchrome	Blanket	Color change to bleached grey with slight green tint.	Same as after 5 hours.	Samples remain flat and square.	Grey with green tint. Samples still flat and square.	Light blue-green color. Semi-rigid. Flat and square.
Saffil Alumina HT	Blanket	Puffed up in thickness. White color and soft texture.	Puffed up in thickness and delaminated. Yellow contamination on bottom side and on setter plate.	White color. Same texture as after 100 hours.	Soft and flexible, irregular surface. Pink color.	Soft and flexible. Pink contamination from Fiberchrome.
Saffil Zirconia HT	Blanket	---	Soft texture. Uneven surface.	At 125 hours: Wavy surface. Edges curled up. Dual layered	At 225 Hours: Off-white color; edges curled up. Off-white color.	At 425 hours: semi-flexible. Irregular surface, edges curled up. Off-white color.

NOTE: 1) Alumina and zirconia blankets charred during start up of furnace (organic binder).

embrittled.

TABLE 6.- VISUAL APPEARANCE OF INSULATION MATERIALS AFTER 1400°C (2552°F) EXPOSURE IN GAS-FIRED KILN

Fiber	Material Type	Visual Observation After 1400°C (2552°F) Exposure			500 Hours
		25 Hours	100 Hours	350 Hours	
Saffil	Felted Fiber	Hard and slightly bowed. Significant shrinkage.	Hard and chalky surface. Large volume contractions.	Board-like with chalky texture. Essentially flat.	Same as after 200 hours
Alumina HT	Cake				
Saffil	Felted Fiber	Off white color with yellow tinge. Slight sticking to setter plate.	Ivory color. Semi-rigid with hard surface.	Board-like and firm. Light yellow color.	Same as after 200 hours
Zirconia HT	Cake	Good condition. No apparent change.	Soft and pliable. Fragile during handling.	Semi-flexible. Edges rounded.	Same as after 200 hours
B&W Mullite	Felted Fiber	Good condition. No apparent change.	Very soft and flexible. Specimen B broke in two during weighing.	Soft and friable. All specimens broken in two (75 hours).	475 hours. Same as after 175 hours.
3 Micron	Felted Fiber	---	Same as after 5 hours	Semi-rigid. Essentially flat.	325 hours. Same as after 175 hours.
Mullite	Cake			Semi-rigid. Sturdy and tough. White color.	
Fiberfrax H	Felted Fiber	No apparent change.	Semi-rigid. Surface texture harder than original material.	Semi-flexible. Bottom flat, edges deformed.	Same as after 200 hours
Kawool	Blanket	White color. Flat and square.	Color change from green-gray to blue-gray.	Semi-rigid. Gray-green color.	Sample has lost nearly all its green color.
Fiberchrome	Blanket	White color with yellow veins. Puffed up. Two distinct layers. Semi-crusty surface.	Puffed up. Traces of chrome contamination from adjacent Fiberchrome.	Soft and pliable. Uneven surface. Two layers. Pink contamination from Fiberchrome.	Pinkish contamination from Fiberchrome.
Saffil	Blanket	Yellow color. Thickness shrunk to 1.3 cm (1/2 inch).	Semi-rigid. Crusty and hard surface texture.	Semi-rigid. Edges curled up. Sticking to setter plate.	Edges curled up. Large shrinkage.
Alumina HT					
Saffil	Blanket		Uneven surface, edges deformed. Yellow color.	Semi-rigid. Edges curled up; center depressed, semi-rigid with crusty surface. Off-white color.	Edges curled up; center depressed, semi-rigid with crusty surface. Off-white color. Severe reduction in thickness.
Zirconia HT					

NOTE: 1) Alumina and zirconia blankets turned black during furnace start up (organic binder).

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TABLE 7.- VISUAL APPEARANCE OF INSULATION MATERIALS AFTER 1600°C (2912°F) EXPOSURE IN GAS-FIRED KILN

Fiber	Material Type	Visual Observation After 1600°C (2912°F) Exposure			
		5 Hours	25 Hours	100 Hours	200 Hours
Saffil Alumina HT	Felted Fiber Cake	Hard surface. Rectangular shape. Large shrinkage.	Severe shrinkage. Rigid and sintered. White Color.	Rigid. Hard crusty surface with chalky texture. Excessive shrinkage.	Vitrified hard surface.
Saffil Zirconia HT	Felted Fiber Cake	Hard crusty surface. Off-white color.	Rigid and brittle. Yellow color. Shrank and deformed into trapezoidal shape with top smaller than bottom.	Crusty and brittle with chalky surface. Off-white color.	Center of fibercake collapsed. Shrinkage more severe on upper surface than lower surface.
B&W Mullite	Felted Fiber Cake	No visual change.	Semi-rigid. Fibers have brittle feel. White color. Little shrinkage.	Very fragile. Specimen broken in two.	Fragile and pliable with very little mechanical strength. Both specimens broken in two.
3 Micron Mullite	Felted Fiber Cake	Very fragile. Crack near end. Retained white color.	Semi-rigid and pliable. White color. Moderate shrinkage. Sticking to setter plate.	Both specimens broken. A broken in two.	Very pliable. No physical integrity. Samples broken into many pieces.
Fiberfrax H	Felted Fiber Cake	Large shrinkage and sample collapse.	Not exposed beyond 5 hrs.	Not exposed beyond 25 hrs.	
Kacwool	Blanket	Large shrinkage. Samples rigid and depressed.	Severe shrinkage and sample collapse	Not exposed beyond 25 hrs.	
Fiberchrome	Blanket	Large shrinkage. Samples rigid and bowed	Severe shrinkage and sample collapse	Not exposed beyond 25 hrs.	
Saffil Alumina HT	Blanket	Semi-hard crust. Edges curled up. Pink discoloration due to Fiberchrome contamination.	Moderate shrinkage. Edges curled up. Rigid. White with pink contamination from Fiberchrome.	Rigid, crusty surface. Edges curled up. Rigid. White with pink discoloration.	Hard surface. Center of sample depressed. Pink discoloration.
Saffil Zirconia HT	Blanket	Hard and crusty. Edges curled up. Off-white color.	Severe shrinkage in thickness. Edges curled up. Rigid. Center of specimen depressed. Yellow color.	Not exposed beyond 25 hrs.	

TABLE 8.— VISUAL APPEARANCE OF INSULATION MATERIALS AFTER EXPOSURE IN ELECTRIC KILN

Fiber	Material Type	Visual Observation After 1000°C (1832°F) Exposure	
		25 Hours	100 Hours
3 Micron Mullite	Felted Fiber Cake	Very fragile. No physical integrity. Specimen cracked through center.	Very soft and flexible. Deforms under own weight. Pulls apart easily. White color.
Fiberfrax H	Blanket	Hard crusty surface. Specimen distorted.	Semi-rigid. Twisted and warped. White color. Hard to measure.
Kaowool	Blanket	Slightly harder than unexposed control.	Soft and flexible. Flat and square. White color.
Fiberchrome	Blanket	Color change from blue-gray to green-gray.	Semi-flexible. Green-gray color. Flat and square.
Saffil Alumina HT	Blanket	Puffed up in height. Layers tend to separate.	Soft and very flexible. Pulls apart easily. Off-white color.
Microquartz	Blanket	Hard crusty surface	Semi-rigid. Uneven surface.
		Visual Observation After 1200°C (2192°F) Exposure for 100 Hours	
3 Micron Mullite	Felted Fiber Cake	Very fragile. White color.	
Fiberfrax H	Blanket	Rigid with deformed edges. Sample B curled and twisted. White color.	
Kaowool	Blanket	Semi-flexible. Reduced thickness. White color.	
Saffil Zirconia HT	Blanket	Semi-rigid on bottom; semi-flexible on top. Beige color.	
Microquartz	Blanket	Rigid with crusty and irregular surface. Excessive shrinkage. White color.	

TABLE 9. - DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 25 HOURS  
AT 1000°C (1832°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Irish Refrasil	Felted Fiber Cake	-2.68	-2.53	-4.2	-3.3	-4.0	-4.0	-5.9	0	11.2	4.6
Saffil Alumina	Felted Fiber Cake	-3.48	-3.94	-3.3	-3.3	-4.0	-4.0	-5.9	-5.9	10.3	9.4
Saffil Zirconia	Felted Fiber Cake	-0.69	-0.44	0	0	0	0	12.5	13.3	-11.9	-14.3
B&W Mullite	Felted Fiber Cake	-0.69	-0.88	0	0	0	0	0	0	-0.7	-0.9
Fiberfrax H	Felted Fiber Cake	-0.28	-0.31	-0.9	-1.7	-4.1	-2.0	-9.1	0	14.9	3.9
Fiberfrax H	Blanket	-0.26	-0.20	-8.1	-11.3	0	0	-27.3	-18.2	49.1	37.7
Kaowool	Blanket	-0.05	-0.28	-3.3	-1.7	0	0	-20.0	-20.0	29.2	27.2
Microquartz	Blanket	-4.80	-4.85	-9.8	-6.6	-12.0	-12.0	-46.7	-60.0	125.0	189.0
Fiberchrome	Blanket	-4.43	-4.54	-1.7	-1.7	0	0	0	0	-3.4	-3.5

NOTE: No sign is increase

- is decrease

TABLE 10. - DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 100 HOURS  
AT 1000°C (1832°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Irish Refrasil	Felted Fiber Cake	-2.50	-2.53	-4.2	-4.2	-4.0	-4.0	-2.4	0	8.4	5.5
Saffil Alumina	Felted Fiber Cake	-2.19	-2.53	-4.2	-4.2	-4.0	-4.0	0	0	6.5	5.7
Saffil Zirconia	Felted Fiber Cake	-0.70	-0.40	0	0	0	0	12.5	6.7	-11.9	-8.7
B&W Mullite	Felted Fiber Cake	-1.16	-0.88	0	0	0	0	-4.0	0	0	-0.9
Fiberfrax H	Felted Fiber Cake	-0.39	-0.41	-0.9	-2.5	-2.0	-2.0	-9.1	-16.7	12.7	25.5
Fiberfrax H	Blanket	-0.29	-0.10	-14.5	-14.5	0	0	-27.3	-27.3	60.0	60.4
Kaowool	Blanket	-0.53	-0.39	-5.0	-3.3	0	0	-20.0	-20.0	30.5	29.3
Microquartz	Blanket	-4.72	-4.47	-9.8	-9.8	-12.0	-12.0	-46.7	-46.7	125.0	110.2
Fiberchrome	Blanket	-4.44	-4.47	-1.7	-1.7	0	0	0	0	-3.4	-3.5

NOTE: No sign is increase  
- is decrease

TABLE 11.- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 500 HOURS  
AT 1000°C (1832°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Irish Refrasil	Felted Fiber Cake	-3.22	-3.43	-4.2	-3.3	-4.0	-4.0	-6.3	0	10.3	2.8
Saffil Alumina	Felted Fiber Cake	-2.81	-3.35	-3.3	-4.2	-4.0	-4.0	-6.3	-6.3	10.3	10.4
Saffil Zirconia	Felted Fiber Cake	-0.47	-0.80	-0.8	0	-2.0	0	12.5	20.0	-10.2	-20.6
B&W Mullite	Felted Fiber Cake	-1.97	-1.67	-3.3	0	0	0	11.1	0	-9.5	-3.0
Fiberfrax H	Felted Fiber Cake	-0.96	-0.90	-4.2	-3.3	-4.1	-2.0	-9.1	-16.7	13.1	22.5
Fiberfrax H	Blanket	-0.52	-0.44	-3.2	-11.3	-4.0	0	-18.2	-18.2	29.1	35.8
Kaowool	Blanket	-0.69	-0.55	-3.3	-1.7	-4.0	-3.8	-20.0	-20.0	26.6	24.5
Fiberchrome	Blanket	-4.30	-4.18	-1.7	-1.7	0	0	0	0	-5.2	-4.3

NOTE: No sign is increase

- is decrease

TABLE 12.- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER EXPOSURE  
AT 1000°C (1832°F) IN ELECTRIC KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
<u>25 HOURS</u>											
3 Micron Mullite	Felted Fiber Cake	-0.94	-1.45	0	0	0	0	7.7	0	-8.0	-9.7
Fiberfrax H	Blanket	0	-0.24	-4.9	-5.0	-3.8	-15.4	0	-20.0	24.0	54.1
Kaowool	Blanket	-0.25	-0.13	-1.6	-1.6	-4.0	0	-33.3	-33.3	58.1	52.7
Fiberchrome	Blanket	-4.31	--	0	--	0	--	0	--	-4.3	--
Saffil Alumina HT	Blanket	-9.69	-10.50	0	0	0	0	66.7	33.3	-45.8	-32.4
Saffil Zirconia HT	Blanket	-8.59	-7.91	0	0	0	-3.8	53.8	53.8	-41.1	-37.3
Microquartz	Blanket	-2.71	-2.67	-4.9	-4.9	-4.0	-4.0	-10.0	-16.7	18.2	28.3
<u>100 HOURS</u>											
3 Micron Mullite	Felted Fiber Cake	-1.58	-2.89	0	0	9.1	13.6	7.7	-8.3	-13.1	-7.0
Fiberfrax H	Blanket	-0.07	-0.37	-1.6	-4.8	-11.5	-11.5	0	-20.0	15.4	47.3
Kaowool	Blanket	-0.34	-0.18	-1.6	-1.6	-2.0	0	-16.7	-16.7	23.5	22.0
Fiberchrome	Blanket	-4.58	--	-1.7	--	-2.0	--	0	--	-0.9	--
Saffil Alumina HT	Blanket	-10.26	-10.87	0	0	0	0	66.7	33.3	-12.0	-32.4
Saffil Zirconia HT	Blanket	-8.93	-8.52	0	0	0	-3.8	53.8	53.8	-41.1	-38.2
Microquartz	Blanket	-2.78	-2.67	-4.9	-4.9	-4.0	-4.0	-10.0	-11.1	18.2	20.8

NOTE: No sign is increase

- is decrease

TABLE 13.— DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 25 HOURS  
AT 1200°C (2192°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Irish Refrasil	Felted Fiber Cake	-2.60	-2.73	-3.3	-3.3	-2.0	-2.0	9.2	29.4	-6.3	-20.9
Safil Alumina HT*	Felted Fiber Cake	-4.73	-1.17	-14.0	-5.0	-16.0	-4.0	-17.6	-2.3	64.2	10.8
Safil Alumina HT*	Felted Fiber Cake	-1.83	--	-8.3	--	-8.0	--	-5.6	--	21.1	--
Safil Zirconia HT	Felted Fiber Cake	-0.86	-0.69	-0.8	-0.8	-2.0	-2.0	30.0	22.2	-21.2	-17.0
B&W Mullite	Felted Fiber Cake	-0.86	-0.76	0.8	0.8	0	0	5.6	0	-6.4	-1.8
Fiberfrax H	Felted Fiber Cake	-0.34	-0.33	-3.4	-4.2	-4.0	-2.0	0	-8.3	7.3	15.6
Fiberfrax H	Blanket	0.18	-0.26	-13.1	-5.7	-8.4	-3.9	-9.1	0	37.9	10.4
Kaowool	Blanket	0.17	-0.27	-0.8	-2.8	0	-2.0	-25.5	-12.3	28.3	18.8
Fiberchrome	Blanket	-4.21	-4.22	-3.3	-3.3	-2.0	-3.9	0	-3.5	0.9	7.0
Safil Alumina HT	Blanket	-8.63	-9.42	-2.5	-2.5	-5.1	-2.0	69.2	50.0	-41.8	-37.3
Safil Zirconia HT	Blanket	-5.66	-5.10	-1.6	-3.3	-6.1	-4.0	0	0	1.1	1.2

\*Sample C After 50 Hours at 1200°C

NOTE: No sign is increase

— is decrease

TABLE 14.- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 100 HOURS  
AT 1200°C (2192°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Irish Refrasiil	Felted Fiber Cake	-2.73	-3.10	-4.1	-4.1	-2.0	-2.0	3.4	17.6	-1.8	-13.9
Saffil Alumina HT	Felted Fiber Cake	-4.64	-1.05	-16.5	-5.0	-16.0	-4.0	-17.6	-8.0	64.2	16.2
Saffil Alumina HT**	Felted Fiber Cake	-1.83	--	-8.3	--	-8.0	--	-5.6	--	21.1	--
Saffil Zirconia HT	Felted Fiber Cake	-0.90	-0.77	-0.8	-1.7	0	-2.0	25.0	16.7	-21.2	-13.4
B&W Mullite	Felted Fiber Cake	-1.27	-1.15	0	0	0	0	0	5.9	-1.8	-8.0
Fiberfrax H	Felted Fiber Cake	-0.67	-0.95	-4.2	-4.2	-4.0	-2.0	-18.2	-16.7	30.3	25.1
Fiberfrax H	Blanket	-0.07	-0.37	-13.1	-5.7	-8.4	-2.0	-18.2	-18.0	51.6	22.3
Kaowool	Blanket	-0.35	-0.92	-2.5	-2.8	-2.0	-2.0	-12.3	-12.3	17.6	16.4
Fiberchrome	Blanket	-4.02	-3.88	-2.5	-2.5	-2.0	-3.9	0	-3.5	-0.9	5.0
Saffil Alumina HT	Blanket	-8.45	-8.96	-2.5	-2.5	-5.1	-4.0	53.8	33.3	-36.7	-29.3
Saffil Zirconia HT*	Blanket	--	--	--	--	--	--	--	--	--	--

\*Inserted at time = 75 hours. No measurement after 100 hour exposure.

\*\*Sample C after 50 hours at 1200°C.

NOTE: No sign is increase  
- is decrease

TABLE 15. - DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 500 HOURS AT 1200°C (2192°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Saffil Alumina HT	Felted Fiber Cake	-5.03	-1.34	-17.4	-6.6	-16.0	-6.0	-17.6	-8.0	67.0	21.6
Saffil Alumina HT**	Felted Fiber Cake	-2.16	--	-10.0	--	-10.0	--	-11.1	--	35.8	--
Saffil Zirconia HT	Felted Fiber Cake	-0.65	-0.45	-2.5	-1.7	-4.0	-4.0	20.0	22.2	-12.1	-14.3
B&W Mullite	Felted Fiber Cake	-2.29	-2.21	-0.8	-0.8	0	0	0	0	0	-1.8
Fiberfrax H	Felted Fiber Cake	-0.92	-0.87	-4.2	-5.0	-4.0	-4.1	-9.1	-8.3	18.5	18.6
Kaowool	Blanket	0	-0.54	-2.5	-2.8	-2.0	-5.9	-21.1	-21.1	32.7	52.3
Fiberchrome	Blanket	-4.47	-4.43	-3.3	-4.1	-3.9	-3.9	-9.1	-12.3	13.5	19.0
Saffil Alumina HT	Blanket	-8.79	-9.42	-4.1	-4.1	-5.1	-6.0	23.1	25.0	-19.0	-20.0
Saffil Zirconia HT*	Blanket	-5.61	-4.94	-4.1	-4.9	-6.1	-4.0	-25.0	-27.8	40.4	45.8

\*After 425 hours at 1200°C.

\*\*Sample C after 450 hours at 1200°C.

NOTE: No sign is increase  
- is decrease

TABLE 16.- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER EXPOSURE  
AT 1200°C (2192°F) IN ELECTRIC KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
<u>25 HOURS</u>											
3 Micron Mullite	Felted Fiber Cake	-1.33	-1.22	--	--	0	0	0	0	--	--
Fiberfrax H	Blanket	-0.09	-0.06	-1.6	-3.3	-3.8	-16.0	-23.1	-8.0	23.6	34.2
Kaowool	Blanket	0.06	-1.11	-4.2	-3.3	-3.8	-3.8	-30.0	-20.0	55.1	33.1
Saffil Zirconia HT	Blanket	-7.44	-7.26	-1.7	-1.7	-4.0	-2.0	-5.6	-5.6	3.5	-2.4
Microquartz	Blanket	-3.45	-3.11	-13.1	-16.7	-20.0	-23.1	-55.0	-55.0	207.8	228.5
<u>100 HOURS</u>											
3 Micron Mullite	Felted Fiber Cake	-2.08	-1.89	--	--	0	0	-10.0	0	--	--
Fiberfrax H	Blanket	-0.09	-0.15	-1.6	-8.3	-5.8	-20.0	-23.1	-12.0	26.4	55.2
Kaowool	Blanket	-0.06	-1.74	-3.3	-1.7	-3.8	-3.8	-30.0	-10.0	53.5	15.5
Saffil Zirconia HT	Blanket	-7.77	-7.59	-3.3	-1.7	-2.0	-4.0	-22.2	-16.7	24.7	10.6
Microquartz	Blanket	-3.38	-3.11	-13.1	-16.7	-16.0	-19.2	-50.0	-50.0	168.6	181.6

NOTE: No sign is increase

- is decrease

TABLE 17. -- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 25 HOURS  
AT 1400°C (2552°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Saffil Alumina HT	Felted Fiber Cake	-2.88	-1.47	-13.3	-5.8	-14.0	-6.0	-17.6	-11.8	59.6	26.5
Saffil Zirconia HT	Felted Fiber Cake	-1.34	-0.79	-1.7	-2.5	-2.0	-4.0	0	0	4.0	7.5
B&W Mullite	Felted Fiber Cake	-3.07	-3.38	0	0	0	0	0	0	-1.8	-1.8
3 Micron Mullite	Felted Fiber Cake	-3.65	-2.94	0	0	0	4.5	-7.7	-7.7	5.4	1.7
Fiberfrax H	Felted Fiber Cake	-0.57	0	-1.7	0.9	-2.1	-4.1	-9.1	-23.1	14.6	43.0
Kaowool	Blanket	-0.51	-0.31	-5.7	-2.5	-2.0	-3.8	-33.3	-27.3	60.3	49.1
Fiberchrome	Blanket	-4.80	-4.58	-3.3	-3.3	-2.0	-2.0	-9.1	-9.1	12.6	12.3
Saffil Alumina HT	Blanket	-8.02	-9.73	-1.7	-3.3	0	-4.0	12.5	30.8	-14.7	-25.0
Saffil Zirconia HT	Blanket	-7.05	-7.15	-12.5	-13.8	-12.0	-10.0	-38.9	-38.9	-101.4	-93.7

NOTE: No sign is increase

- is decrease

TABLE 18.- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 100 HOURS  
AT 1400°C (2552°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Saffil Alumina HT	Felted Fiber Cake	-2.92	-1.43	-13.3	-5.0	-8.0	-6.0	-17.6	-11.8	56.1	25.7
Saffil Zirconia HT	Felted Fiber Cake	-1.14	-0.50	-2.5	-2.5	-4.0	-4.0	0	0	7.1	8.3
B&W Mullite	Felted Fiber Cake	-4.47	-5.06	-1.7	-1.7	-2.0	-2.0	-5.9	-5.9	6.3	6.2
3 Micron Mullite*	Felted Fiber Cake	-5.65	-6.33	---	---	0	4.5	-15.4	-23.1	---	---
Fiberfrax H	Felted Fiber Cake	-1.15	-0.16	-3.4	-4.2	-4.2	-4.1	-9.1	-23.1	18.2	43.0
Kaowool	Blanket	-0.73	-0.50	-4.1	-2.5	-2.0	-3.8	-41.7	-36.4	82.8	69.6
Fiberchrome	Blanket	-5.38	-5.15	-2.5	-3.3	-3.9	-2.0	-9.1	-9.1	12.6	12.3
Saffil Alumina HT	Blanket	-8.16	-9.73	-1.7	-3.3	0	-2.0	0	23.1	-4.4	-19.7
Saffil Zirconia HT	Blanket	-7.05	-7.08	-13.3	-13.0	-14.0	-12.0	-50.0	-44.4	154.3	122.2

NOTE: No sign is increase  
- is decrease

\*After 75 hours at 1400°C.

TABLE 19.- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 500 HOURS AT 1400°C (2552°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (g)		Length (in.)		Width (in.)		Thickness (in.)		Density (g/cc)	
		A	B	A	B	A	B	A	B	A	B
Saffil Alumina HT	Felted Fiber Cake	-2.83	-1.47	-16.7	-7.5	-16.0	-8.0	-17.6	-11.8	70.2	31.9
Saffil Zirconia HT	Felted Fiber Cake	-1.26	-1.00	-11.0	-11.7	-12.0	-12.0	-18.8	-12.5	57.1	47.5
B&W Mullite	Felted Fiber Cake	-7.37	-8.57	-3.3	-3.3	-4.0	-4.0	-11.8	-17.6	14.4	21.2
3 Micron Mullite*	Felted Fiber Cake	-10.30	-12.36	--	--	--	--	-23.1	-23.1	--	--
Fiberfrax H	Felted Fiber Cake	-1.81	-0.53	-10.3	-11.9	-12.5	-14.3	-27.3	-30.8	68.2	92.4
Kaowool	Blanket	-1.07	-0.50	-9.0	--	-12.0	-15.4	-50.0	-45.5	150.0	--
Fiberchrome	Blanket	-6.66	-6.36	-5.8	-5.8	-5.9	-4.0	-18.2	-18.2	31.5	28.9
Saffil Alumina HT	Blanket	-8.38	-9.49	-1.7	-4.2	0	-4.0	-12.5	7.7	8.8	-7.9
Saffil Zirconia HT	Blanket	-7.17	-6.74	-20.8	-21.1	-20.0	-20.0	-55.6	-55.6	235.7	238.1

\* Broken samples. Weight loss is questionable due to possible loss of material.

NOTE: No sign is increase  
- is decrease

TABLE 20.- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 25 HOURS  
AT 1400°C (2552°F) IN ELECTRIC KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	E	A	B	A	B	A	P
Saffil Alumina HT	Felted Fiber Cake- New Fiber	-0.36	-0.40	-1.7	-0.8	-2.0	-2.0	-4.8	-4.8	8.5	7.4
	Felted Fiber Cake- Old Fiber	-3.80	---	-11.7	---	-12.0	---	-16.7	---	48.6	--
Saffil Alumina HT	New Blanket	-8.92	-7.22	-3.3	-5.0	-4.0	-4.0	18.8	18.8	-16.9	-14.8
	Old Blanket	-10.71	-9.64	-2.4	-3.3	-2.0	-2.0	33.3	41.7	-29.5	-32.5
Saffil Alumina HT	New Blanket	-6.60	-6.75	-3.3	-3.3	-8.0	-12.0	-50.0	-37.5	109.4	72.0
	Old Blanket	-7.39	---	-5.0	---	-8.0	---	-28.6	---	48.6	--

NOTE: No sign is increase

- is decrease

TABLE 21.— DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 25 HOURS AT 1600°C (2912°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
Saffil Alumina HT	Felted Fiber Cake	-1.47	-4.59	-21.7	-25.0	-22.0	-26.0	-23.5	-27.8	111.1	138.5
Saffil Zirconia HT	Felted Fiber Cake	0.12	0.62	-10.2	-8.3	-12.0	-12.0	-33.3	-29.4	90.3	76.7
B&W Mullite	Felted Fiber Cake	-8.92	-7.29	--	-2.5	-4.0	-4.0	-22.2	-11.1	--	83.2
3 Micron Mullite *	Felted Fiber Cake	--	--	--	--	--	--	--	--	--	--
Fiberfrax H* *	Felted Fiber Cake	0.40	0.74	-27.5	-25.9	-32.0	-32.7	-30.0	-36.4	202.5	216.8
Kaowool	Blanket	-0.33	--	-10.0	--	-20.0	--	-50.0	--	176.2	--
Fiberchrome	Blanket	-5.79	--	-20.8	--	-28.0	--	-50.0	--	230.4	--
Saffil Alumina HT	Blanket	-11.22	-11.25	-5.8	-5.0	-12.0	-12.0	-16.7	-28.6	28.4	49.3
Saffil Zirconia HT	Blanket	-6.37	-9.14	-16.4	--	-12.0	-16.0	-50.0	-50.0	154.2	--

\* Samples broken into pieces.

\*\* After 5 hours at 1600°C

NOTE: No sign is increase  
- is decrease

TABLE 22.— DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 100 AND 200 HOURS  
AT 1600°C (2912°F) IN GAS-FIRED KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		A	B	A	B	A	B	A	B	A	B
<u>100 HOURS</u>											
Safil Alumina HT	Felted Fiber Cake	-1.34	-4.51	-24.2	-27.5	-26.0	-28.0	-23.5	-33.3	129.9	175.2
Safil Zirconia HT	Felted Fiber Cake	-0.20	0.41	-16.9	-12.5	-20.0	-20.0	-44.4	-41.2	165.5	144.0
B&W Mullite	Felted Fiber Cake	-10.64	-8.68	--	-1.7	-6.0	-4.0	-22.2	-16.7	--	15.9
3 Micron Mullite	Felted Fiber Cake	--	-9.33	--	--	--	-6.4	--	-23.1	--	--
Safil Alumina HT	Blanket	-10.34	-11.26	-8.3	-8.3	-12.0	-12.0	-25.0	-16.7	45.9	53.7
<u>200 HOURS</u>											
Safil Alumina HT	Felted Fiber Cake	-1.22	-4.51	-25.0	-28.3	-26.0	-28.0	-23.5	-33.3	133.3	178.0
Safil Zirconia HT	Felted Fiber Cake	-1.01	-0.20	-18.6	-16.7	-28.0	-24.0	-44.4	-45.5	202.7	198.3
B&W Mullite	Felted Fiber Cake	-14.75	-11.12	--	--	-6.0	-8.0	-27.8	-27.8	--	--
3 Micron Mullite	Felted Fiber Cake	--	-11.15	--	--	--	-14.0	--	-38.5	--	--
Safil Alumina HT	Blanket	-11.31	-11.77	-9.2	-8.3	-16.0	-16.0	-33.3	-16.7	73.0	61.2

NOTE: No sign is increase  
- is decrease

TABLE 23.— COMPARISON OF DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 25 AND 100 HOURS IN GAS-FIRED (G) AND ELECTRIC (E) KILN

Fiber	Material Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
		G	E	G	E	G	E	G	E	G	E
<u>25 Hours at 1000°C</u>											
Fiberfrax H	Blanket	-0.23	-0.12	-9.7	-5.0	0	-9.6	-22.7	-10.0	43.9	39.0
Kaowool	Blanket	-0.17	-0.19	-2.5	-1.6	0	-2.0	-20.0	-33.3	28.2	55.4
Fiberchrome	Blanket	-4.48	-4.31	-1.7	0	0	0	0	0	-3.5	-4.3
Microquartz	Blanket	-4.82	-2.69	-8.2	-4.9	-12.0	-4.0	-53.3	-13.3	157.0	23.2
<u>100 Hours at 1000°C</u>											
Fiberfrax H	Blanket	-0.20	-0.22	-14.5	-3.2	0	-11.5	-27.3	-10.0	60.2	31.4
Kaowool	Blanket	-0.46	-0.26	-4.2	-1.6	0	-1.0	-20.0	-16.7	29.9	22.8
Fiberchrome	Blanket	-4.45	-4.58	-1.7	-1.7	0	-2.0	0	0	-3.5	-0.9
Microquartz	Blanket	-4.60	-2.72	-9.8	-4.9	-12.0	-4.0	-46.7	-10.5	117.6	19.5
<u>25 Hours at 1200°C</u>											
Fiberfrax H	Blanket	-0.04	-0.07	-9.4	-2.5	-6.2	-9.9	-4.6	-15.5	24.2	28.9
Kaowool	Blanket	-0.05	-0.53	-1.8	-3.8	-1.0	-3.8	-18.8	-25.0	23.5	44.1
Saffil Zirconia HT	Blanket	-6.64	-7.35	-2.5	-1.7	-5.0	-3.0	0	-5.6	1.1	3.0
<u>100 Hours at 1200°C</u>											
Fiberfrax H	Blanket	-0.22	-0.12	-9.4	-5.0	-5.2	-12.9	-18.1	-17.5	37.0	40.8
Kaowool	Blanket	-0.64	-0.90	-2.6	-2.5	-2.0	-3.8	-12.3	-20.0	17.0	34.5

NOTE: No sign is increase

- is decrease

Values are average for two samples.

TABLE 24 WEIGHT LOSS AND DIMENSIONAL CHANGE SUMMARY FOR 1000°C (1832°F) EXPOSURE\*

Percent Change	Length	Width	Thickness	Weight
Increase or 0-2% Decrease	Saffil Zirconia HT Cake B&W Mullite Cake 3 Micron Mullite Cake Fiberchrome Blanket Saffil Alumina HT Blanket Saffil Zirconia HT Blanket	Saffil Zirconia HT Cake B&W Mullite Cake 3 Micron Mullite Cake Fiberchrome Blanket Saffil Alumina HT Blanket Saffil Zirconia HT Blanket	Saffil Zirconia HT Cake B&W Mullite Cake 3 Micron Mullite Cake Fiberchrome Blanket Saffil Alumina HT Blanket Saffil Zirconia HT Blanket	Saffil Zirconia HT Cake Fiberfrax H Cake Kaowool Blanket Fiberfrax H Blanket B&W Mullite Cake
2-6% Decrease	Irish Refrasil Cake Saffil Alumina HT Cake Fiberfrax H Cake Kaowool Blanket	Irish Refrasil Cake Saffil Alumina HT Cake Fiberfrax H Cake Kaowool Blanket	Irish Refrasil Cake Saffil Alumina HT Cake Fiberfrax H Cake Kaowool Blanket	$\frac{2-4\%}{3}$ Micron Mullite Cake Irish Refrasil Cake Saffil Alumina HT Cake
Greater than 6% Decrease	Fiberfrax H Blanket Microquartz Blanket	Microquartz Blanket	Fiberfrax H Cake Kaowool Blanket Fiberfrax H Blanket Microquartz Blanket	$\frac{4-6\%}{4}$ Fiberchrome Blanket Microquartz Blanket

\*500 hours of actual exposure or 100 hours extrapolated to 500 hours.

TABLE 25 WEIGHT LOSS AND DIMENSIONAL CHANGE SUMMARY FOR 1200°C (2192°F) EXPOSURE \*

Percent Change	Length	Width	Thickness	Weight
Increase or 0-2% decrease	Saffil Zirconia HT Cake B&W Mullite Cake 3 Micron Mullite Cake	B&W Mullite Cake 3 Micron Mullite Cake	Irish Refrasil Cake Saffil Zirconia HT Cake Saffil Alumina HT Blanket B&W Mullite Cake	Saffil Zirconia HT Cake Fiberfrax H Cake Fiberfrax H Blanket Kaowool Blanket
2-6% Decrease	Irish Refrasil Cake Fiberfrax H Cake Kaowool Blanket Fiberchrome Blanket Saffil Alumina HT Blanket Saffil Zirconia HT Blanket		Irish Refrasil Cake Saffil Zirconia HT Cake Fiberfrax H Cake Kaowool Blanket Fiberchrome Blanket Saffil Zirconia HT Blanket	2-4% Irish Refrasil Cake Saffil Alumina HT Cake B&W Mullite Cake 3 Micron Mullite Cake 4-6% Fiberchrome Blanket
Greater than 6% Decrease	Saffil Alumina HT Cake Fiberfrax H Blanket	Saffil Alumina HT Blanket Fiberfrax H Blanket	Saffil Alumina HT Cake Saffil Alumina HT Blanket Fiberfrax H Blanket Kaowool Blanket Fiberchrome Blanket Saffil Zirconia HT Blanket	Saffil Alumina HT Blanket Saffil Zirconia HT Blanket

\*500 hours of actual exposure or 100 hours extrapolated to 500 hours.

TABLE 26 WEIGHT LOSS AND DIMENSIONAL CHANGE SUMMARY FOR 1400°C (2552°F) EXPOSURE\*

Percent Change	Length	Width	Thickness	Weight
Increase or 0-2% Decrease		Saffil Alumina HT Blanket		Saffil Zirconia HT Cake Fiberfrax H Cake Kaowool Blanket
2-6% Decrease	B&W Mullite Cake 3 Micron Mullite Cake Kaowool Blanket Fiberchrome Blanket Saffil Alumina HT Blanket	B&W Mullite Cake 3 Micron Mullite Cake <b>Fiberchrome Blanket</b>	Saffil Alumina HT Blanket	<u>2-4%</u> Saffil Alumina HT Cake
Greater than 6% Decrease	Saffil Alumina HT Cake Saffil Zirconia HT Cake Fiberfrax H Cake Saffil Zirconia HT Blanket	Saffil Alumina HT Cake Saffil Zirconia HT Cake Fiberfrax H Cake Kaowool Blanket Saffil Zirconia HT Blanket	Saffil Alumina HT Cake Saffil Zirconia HT Cake B&W Mullite Cake 3 Micron Mullite Cake Fiberfrax H Cake Kaowool Blanket Fiberchrome Blanket Saffil Zirconia HT Blanket	B&W Mullite Cake 3 Micron Mullite Cake Fiberchrome Blanket Saffil Alumina HT ** Blanket Saffil Zirconia HT ** Blanket

\*\* Weight loss due to organic binder. No change in weight loss between 25 hour and 500 hours exposure.

\* 500 hours of actual exposure of 100 hours extrapolated to 500 hours.

TABLE 27 WEIGHT LOSS AND DIMENSIONAL CHANGE SUMMARY FOR 1600°C (2912°F) EXPOSURE \*

Percent Change	Length	Width	Thickness	Weight
Increase or 0-2% Decrease				Saffil Zirconia HT Cake
2-6% Decrease				<u>2-4%</u> Saffil Alumina HT Cake
Greater than 6% Decrease	Saffil Alumina HT Cake Saffil Zirconia HT Cake B&W Mullite Cake 3 Micron Mullite Cake Saffil Alumina HT Blanket	Saffil Alumina HT Cake Saffil Zirconia HT Cake B&W Mullite Cake 3 Micron Mullite Cake Saffil Alumina HT Blanket	Saffil Alumina HT Cake Saffil Zirconia HT Cake B&W Mullite Cake 3 Micron Mullite Cake Saffil Alumina HT Blanket	B&W Mullite Cake 3 Micron Mullite Cake Saffil Alumina HT Blanket

\*500 hours of actual exposure or 100 hours extrapolated to 500 hours.

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TABLE 28 SUMMARY OF SEM OBSERVATIONS AFTER FURNACE EXPOSURE\*

Fiber	Material Type	Scanning Electron Microscopy Observations			1600°C (2912°F)
		1000°C (1832°F)	1200°C (2192°F)	1400°C (2552°F)	
Irish Refrassil	Felted Fiber Cake	No Change	100 hrs - No change	--	--
Saffil Alumina HT	Felted Fiber Cake	No change	Grain growth and fiber sintering	Grain growth and sintering. Severe embrittlement.	200 hrs. Severe grain growth and fiber fusion.
Saffil Zirconia HT	Felted Fiber Cake	No change	No change	Extensive grain growth and fiber embrittlement	200 hrs. Severe grain growth and evidence of fiber fusion.
B&W Mullite	Felted Fiber Cake	No change	No change	Grain growth of varying severity in most fibers.	200 hours. Severe grain growth in all fibers and embrittlement.
3 Micron Mullite	Felted Fiber Cake	100 hrs - No change	100 hrs - No change	Moderate grain growth and fiber embrittlement.	200 hrs. Severe grain growth and fiber fusion.
Fiberfrax H	Felted Fiber Cake	No change	No change	Fiber embrittlement and grain growth in some fibers.	5 hrs. Severe grain growth and fiber fusion.
Fiberfrax H	Blanket	No change	100 hrs. Some fiber embrittlement.	--	--
Kaowool	Blanket	No change	No change	Moderate grain growth	5 hrs. Severe grain growth with fiber fusion.
Fiberchrom	Blanket	No change	No change	Extensive grain growth with fiber fusion.	5 hrs. Severe fiber fusion and consolidation.
Saffil Alumina HT	Blanket	100 hours - No change	Slight grain growth	Grain growth but no fiber embrittlement	200 hrs. Severe grain growth and fiber sintering.
Saffil Zirconia HT	Blanket	100 hrs - No change	No change	Moderate grain growth, fiber fusion and embrittlement.	25 hrs. Severe grain growth with fiber sintering
Microquartz	Blanket	100 hours - No change	--	--	--

\*500 hour exposure unless otherwise specified.

TABLE 29.- X-RAY DIFFRACTION SUMMARY FOR FIBERCAKES AND BLANKET INSULATION

Material	Control	1000°C	1200°C	1400°C	1600°C
Irish Refrasil Type	Noncrystalline	Noncrystalline after 500 hours	Moderately crystalline (Alpha Crystobalite) after 50 hrs. Strongly crystalline after 100 hrs.		
White Quartz Blanket	Noncrystalline	Low intensity mixture of Crystobalite and Alpha Quartz after 50 hrs. Increased crystallinity with greater Crystobalite content and reduced Alpha Quartz content after 100 hrs.			
Fiberchrome Blanket	Noncrystalline	Weak Mullite/Sillimanite peak after 100 hrs. Three weak Mullite/Sillimanite peaks after 500 hrs.	Weak Crystobalite peak (4.05A°) and Mullite/Sillimanite peaks after 500 hrs.	Strong Crystobalite peaks and moderately strong Mullite peaks after 500 hrs.	Strong Mullite peaks after 5 hrs. No evidence of Crystobalite.
Wool Ceram	Noncrystalline	Weak Mullite peaks after 500 hrs.	Increased Mullite intensity and weak Crystobalite peak after 500 hrs.	Mullite peaks after 500 hrs are of same intensity as after 500 hrs at 1000°C. No evidence of Crystobalite.	Moderately strong Mullite peaks after 5 hrs.
Fiberfrax Cake	Noncrystalline	Weak Mullite peaks after 500 hrs.	Weak Mullite peaks after 500 hrs. Same as after 500 hrs at 1000°C	Moderately strong Mullite peaks after 500 hrs.	Moderately strong peaks identifiable as Mullite and Sillimanite after 5 hrs.
Fiberfrax Blanket	Noncrystalline	Moderately strong Mullite peaks after 500 hrs.	Moderately strong Mullite peaks after 50 hrs. Mullite and trace unknowns after 100 hrs.		
B&W Mullite Cake	3 weak unidentified lines.	Weak Mullite peaks after 500 hrs.	Moderately strong Mullite peaks after 500 hrs.	Strongly crystalline after 500 hrs. Phases present are Mullite and a considerable amount of Alpha Alumina (Corundum). Strong peak at 3.18 A° indicative of a triclinic kyanite structure.	Strongly crystalline after 200 hrs. Phases present are Mullite and a considerable amount of Alpha Alumina (Corundum) in same relative proportions as after 500 hrs at 1400°C. Very weak peak at 3.18 A°.
3 Micron Mullite Cake	Very weak Mullite peaks.	Weak Mullite peaks after 100 hrs.	Moderately strong Mullite peaks and trace unknowns after 100 hrs.	Moderately strong crystalline peaks after 500 hrs. Principally Mullite with some Alpha Alumina (Corundum).	Moderately strong crystalline peaks after 200 hrs. Mullite and Alpha Alumina (Corundum) in about equal parts.
Saffil Alumina HT Blanket	5 weak unidentified peaks. Major peak at about 1.395 A°, indicative of Gamma Alumina (Cubic).	8 weak unidentified peaks after 100 hrs. Major peak at about 1.395 A°, indicative of Gamma Alumina.	Moderately strong Alpha Alumina (Corundum) peaks after 500 hrs.	Moderately strong Alpha Alumina (Corundum) peaks after 500 hrs.	Moderately strong Alpha Alumina (Corundum) peaks after 25 hrs. Strong Corundum peaks and weak Mullite peaks after 200 hrs.
Saffil Alumina HT Cake	5 weak unidentified peaks.	6 weak unidentified peaks after 500 hrs. Major peak at about 1.395 A° indicative of Gamma Alumina.	Moderately strong Alpha Alumina (Corundum) peaks and weak Mullite peaks after 500 hrs.	Strong Alpha Alumina (Corundum) peaks and weak Mullite peaks after 500 hrs.	Very strong Alpha Alumina (Corundum) peaks and moderately strong Mullite peaks after 25 hrs and after 200 hrs.
Saffil Zirconia HT Blanket	Strongly crystalline crystal form appears to be tetragonal or mixture of cubic and tetragonal.	Strongly crystalline after 100 hrs, similar to control.	Strongly crystalline after 425 hrs, similar to control.	Moderately strong crystalline peaks after 500 hrs. Crystal form appears to be mixture tetragonal and monoclinic (Baddeleyite).	Strongly crystalline after 15 hrs. Crystal form appears to be mixture of cubic and tetragonal with a trace of monoclinic (Baddeleyite).
Saffil Zirconia HT Cake	Strong crystalline. Crystal form appears to be mixture of cubic and tetragonal.	Strongly crystalline after 500 hrs. Crystal form appears to be tetragonal.	Very strongly crystalline after 500 hrs. Crystal form appears to be tetragonal.	Strongly crystalline after 500 hrs. Crystal form appears to be a mixture of cubic, tetragonal and monoclinic (Baddeleyite).	Strongly crystalline after 25 hrs. Crystal form appears to be tetragonal with a trace of monoclinic. Very strongly crystalline after 200 hrs. Crystal form appears to be tetragonal with a moderate amount of monoclinic.

TABLE 30.-ROOM TEMPERATURE THERMAL CONDUCTIVITY AND DENSITY OF BLANKET MATERIALS

Exposure/Material	Density			Thermal Conductivity		
	g/cc	% Change	W/m-°K	Btu-in./hr-ft <sup>2</sup> -°F	% Change	
Controls						
Saffil Alumina HT	.076	N/A	.0449	.312	N/A	
Kaowool 1400	.141	N/A	.0374	.260	N/A	
Fiberchrome	.106	N/A	.0386	.268	N/A	
500 Hr @ 1000 °C						
Saffil Alumina HT	.047*	-37*	.0300*	.208*	-1*	
Kaowool 1400	.189	34	.0484	.336	29	
Fiberchrome	.110	4	.0305	.212	-21	
500 hr @ 1200 °C						
Saffil Alumina HT	.062	-18	.0438	.304	-3	
Kaowool 1400	.193	37	.0478	.332	28	
Fiberchrome	.122	15	.0403	.280	4	
500 hr @ 1400 °C						
Saffil Alumina HT	.072	-5	.0743	.516	65	
Kaowool 1400	.275	95	.0588	.408	57	
Fiberchrome	.146	38	.0651	.452	69	

\*100-hr exposure.

NOTE: No sign is increase  
 - is decrease

TABLE 31.- DIMENSIONAL AND WEIGHT CHANGE OF DENSITY/SHRINKAGE SAMPLES AFTER 25 CYCLES AT 1200°C  
(2192°F) AND 1400°C (2552°F) UNDER RADIANT HEATING

Fiber	Material	Type	Weight (%)		Length (%)		Width (%)		Thickness (%)		Density (%)	
			A	B	A	B	A	B	A	B	A	B
<u>1200°C</u>												
Saffil Alumina	HT	RSI	-0.03	-0.06	0.9	0.9	0	0	5.3	0	-0.7	-1.3
Saffil Zirconia	HT	RSI	-0.24	-0.43	0	0	0	0	0	-6.3	0	6.0
B&W Mullite		RSI	-0.66	-0.23	0	0	2.3	0	0	0	-2.5	0
<u>1400°C</u>												
Saffil Alumina	HT	RSI	-0.06	0.03	0	0	0	-0.8	0	0	0.7	0.7
Saffil Zirconia	HT	RSI	-0.32	-0.23	0	0	-0.9	0	0	0	0	0
B&W Mullite		RSI	-0.42	-0.29	-0.3	-0.9	0	-2.2	0	0	0	2.6

NOTE: No sign is increase  
- is decrease

TABLE 32.- FLEXURE TEST RESULTS FOR RSI MATERIAL

Material/Exposure	Flexural Strength		Modulus of Elasticity N/m <sup>2</sup>	Strain at Failure (%)
	psi	N/m <sup>2</sup>		
Saffil Alumina HT	76.3	526 x 10 <sup>3</sup>	32,000	0.28
	71.9	496 x 10 <sup>3</sup>	29,000	0.29
	76.0	524 x 10 <sup>3</sup>	32,000	0.29
Saffil Zirconia HT	68.3	471 x 10 <sup>3</sup>	31,000	0.24
	73.2	505 x 10 <sup>3</sup>	31,000	0.26
	93.6	645 x 10 <sup>3</sup>	36,000	0.29
B&W Mullite	31.5	217 x 10 <sup>3</sup>	6,500	0.54
	30.3	209 x 10 <sup>3</sup>	6,100	0.53
	30.7	212 x 10 <sup>3</sup>	7,200	0.47

TABLE 33.- THERMAL CONDUCTIVITY OF RSI MATERIALS

Material	Room Temperature Thermal Conductivity		1200°C Exposure		1400°C Exposure	
	Btu-in./hr-ft <sup>2</sup> -°F	W/m-°K	Btu-in./hr-ft <sup>2</sup> -°F	W/m-°K	Btu-in./hr-ft <sup>2</sup> -°F	W/m-°K
Saffil Alumina HT	1.18	0.170	0.97	0.140	0.95	0.137
Saffil Zirconia HT	0.36	0.052	0.38	0.055	0.36	0.052
B&W Mullite	0.48	0.069	0.42	0.060	0.40	0.058

TABLE 34.- COST SUMMARY FOR 930 m<sup>2</sup> (10,000 ft<sup>2</sup>) OF RSI TILE

	Hours	Burdened Dollars
<b>Labor</b>		
Supervision	2,000	48,660
Fabrication	12,000	211,965
Quality Control	2,000	41,501
Shipping	384	9,257
Contract Support	200	4,441
<b>TOTAL LABOR</b>	<b>16,584</b>	<b>315,824</b>
<b>Materials</b>		
Alumina Fiber		124,450
Silbond H-4		12,288
Isopropyl Alcohol		10,794
NH <sub>4</sub> OH		301
Separan AP-30		569
<b>TOTAL MATERIALS</b>		<b>148,402</b>
<b>FREIGHT</b>		<b>2,256</b>
<b>TOTAL LABOR &amp; MATERIALS</b>		<b>466,482</b>
<b>PROFIT</b>		<b>46,648</b>
<b>TOTAL PRICE</b>		<b>513,130</b>